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Ionic liquid-anionic surfactant based aqueous two-phase extraction for determination of antibiotics in honey by high-performance liquid chromatography

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ABSTRACT

An ionic liquid-anionic surfactant based aqueous two-phase extraction was developed and applied for the extraction of tetracycline, oxytetracycline and chloramphenicol in honey. The honey sample was mixed with Na_2EDTA aqueous solution. The sodium dodecyl sulfate, ionic liquid 1-octyl-3-methylimidazolium bromide and sodium chloride were added in the mixture. After the resulting mixture was ultrasonically shaken and centrifuged, the aqueous two phase system was formed and analytes were extracted into the upper phase. The parameters affecting the extraction efficiency, such as the volume of ionic liquid, the category and amount of salts, sample pH value, extraction time and temperature were investigated. The limits of detection of tetracycline, oxytetracycline and chloramphenicol were 5.8, 8.2 and 4.2 $\mu g\ kg^{-1}$, respectively. When the present method was applied to the analysis of real honey samples, the recoveries of analytes ranged from 85.5 to 110.9% and relative standard deviations were lower than 6.9%.

1. Introduction

Honey is a kind of directly consumed food. It is a conglomeration of several organic and inorganic constituents, with glucose and fructose contributing about 75% of the total. Other organic constituents include di/tri/oligo saccharides, aliphatic acids, vitamins, amino acids and proteins and inorganic constituents, including water, potassium, sodium, calcium, magnesium, copper, manganese, iron, chloride, sulfur, phosphorus and silica [1]. Antibiotics such as tetracycline (TC), oxytetracycline (OTC) and chloramphenicol (CAP) are used in beekeeping to fight against bacterial diseases, among which the worst ones are the American Foulbrood (AFB) and the European Foulbrood (EFB) which destroy honeybee larvae [2]. Consequently, such antibiotics may persist at trace levels in honey, being a potential risk to honey consumers in terms of possible allergic reactions, liver damage, yellowing of teeth, gastro-intestinal disturbance, aplastic anemia and the development of bacterial resistance [3]. Therefore, reliable analytical techniques are increasingly demanded to detect antibiotic residues in honey.

Some countries have established maximum residue limits (MRLs) for tetracycline antibiotics in honey, while others do not tolerate any residue level. For instance, Codex Alimentarius and the European Union (EU) have not established MRL for veterinary medicine in honey [4,5]. In China, the MRL for the total tetracycline antibiotics in honey should not exceed 50 $\mu g\ kg^{-1}$ [6]. The Spanish plan for residue control and healthy food (Plan CRE HA) has established the MRL of 100 $\mu g\ kg^{-1}$ for TC, OTC and chlortetracycline (CTC) in honey [7]. In Brazil the National Program for Honey Residues Control established by the Ministry of Agriculture has provided that the MRL for TC, OTC and CTC should not exceed 200 $\mu g\ kg^{-1}$ [8]. China, the European Commission, the United States and some other countries have strictly banned the use of CAP in food-producing animals.

TC, OTC and CAP were successfully determined by high-performance liquid chromatography (HPLC) in the reversed-phase mode, with different detection modes, such as ultraviolet [9,10,11], fluorescence [12], chemiluminescence [13] and mass spectrometry [14,15]. Furthermore, extraction and preconcentration procedures are very important before instrumental analysis. The liquid-liquid extraction (LLE) [16] and solid-phase extraction

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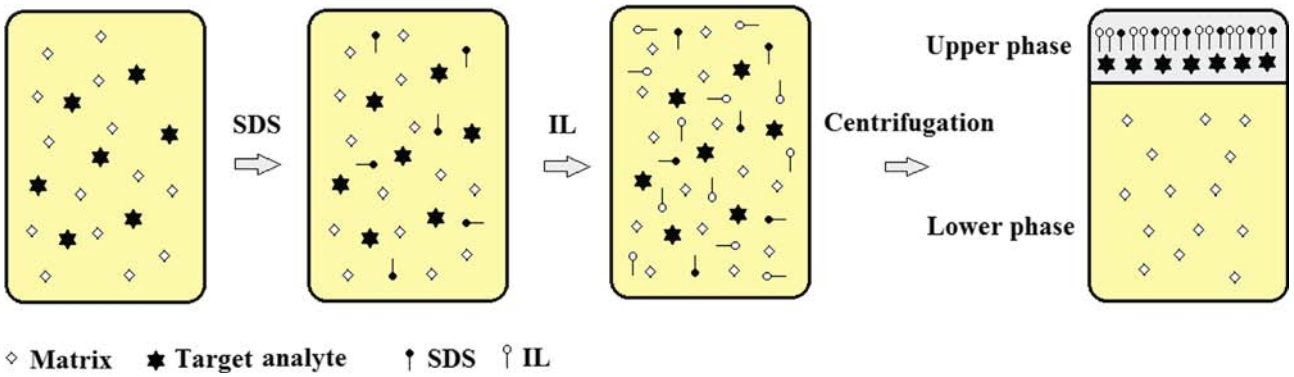

Fig. 1. Aqueous two-phase extraction.

(SPE) [17,18] have been extensively applied to the pretreatment of liquid food samples. However, most of these methods are time consuming and usually toxic due to the use of organic solvents.

When cationic and anionic surfactants in aqueous solution are mixed at an appropriate molar ratio, two immiscible aqueous phases with a clear interfacial boundary can be formed [19]. Aqueous two-phase (ATP) was first discovered by Karler, et al. in 1989 [20]. They reported the spontaneous formation of vesicles by mixing a cationic surfactant cetyltrimethylammonium tosylate (CTAT) and an anionic surfactant sodium dodecylbenzene sulfonate (SDBS) in aqueous solution. The hydrophobic and electrostatic interactions are regarded as the main driving forces for the spontaneous formation of vesicles [21]. Aqueous mixtures of anionic and cationic surfactants exhibit rich microstructured phase behavior and many unique properties that arise from stronger electrostatic interactions than single pure surfactant does and are often more surface active than either pure surfactant [22]. There are a wide variety of microstructures, such as spherical and rodlike micelles, vesicles, lamellar phase and flat discs, etc [23]. The formation of ATP can be attributed to the coexistence of different sized micelles in the upper and lower phase [24].

In the study, the ATP was formed in the presence of anionic surfactant sodium dodecyl sulfate (SDS) and ionic liquid (IL) 1-octyl-3-methylimidazolium bromide (O[MIM]Br) which is surface active according to a hydrophobic tail and a hydrophilic headgroup. When the molar ratio of the two surfactants changes, two kinds of ATPs are observed. One is SDS-rich ATP which means that the concentration of SDS is higher than that of IL and the other one is IL-rich which means that the concentration of IL is higher than that of SDS. When the ATP is formed, the upper phase is surfactant-rich which forms lamellar micelles and the lower phase is surfactant-dilute with spherical micelles. Analytes are extracted into the surfactant-rich phase according to the different distribution coefficients of them in the two phases, as can be seen in Fig. 1. The ionic liquid-anionic surfactants based aqueous two-phase extraction (IL/AS-based ATPE) is an easy and rapid sample pretreatment method. The present method is considered to be environmentally friendly because of the avoidance of traditional volatile solvents. Compared with traditional IL/salt ATPE, the amount of ionic liquid and salts used to form ATP is smaller and the phase separation time is shorter.

2. Experimental

2.1. Instrumentations

The 1100 series liquid chromatography (Agilent Technologies, Palo Alto, USA) equipped with UV detector and quaternary gradient pump was used. Zorbax Eclipse XDB-C18 column (4.6 mm × 150 mm, 3.5 μm) and a C18 guard column (7.5 mm × 2.1 mm, 5 μm) were used. Ultrasonic mixing was performed with a 100 W ultrasonic cleaner (model KQ-100DE, Kunshan Ultrasonic Instrument, Kunshan, China). The phase separation was performed on HC-2066 high-speed centrifuge (Anhui USTC Zonkia Scientific Instruments, Hefei, China).

2.2. Reagents and chemicals

TC, OTC and CAP were purchased from the National Institute for the Control of Pharmaceutical and Biological Products (Beijing, China). The chemical structures of the compounds are shown in Fig. 2. O[MIM]Br was purchased from Shanghai Chengjie Chemical Reagent Co. Ltd. SDS was purchased from Sinopharm Chemical Reagent (Shanghai, China). Chromatographic grade methanol was purchased from Fisher Scientific Company (Loughborough, UK) and pure water was obtained with a Milli-Q water purification system (Millipore, Billerica, USA). All other chemicals used, such as formic acid, hydrochloric acid and $Na_2EDTA{\bullet}2H_2O$, were of analytical grade.

2.3. Standard solutions

The individual stock solution of each analyte (1000 μg mL^{-1}) was prepared by dissolving 10 mg of the analyte in 10 mL of methanol and stored in the refrigerator at 4 °C. The mixed stock solution containing all analytes (100 μg mL^{-1}) was prepared with individual stock solutions by diluting with methanol and stored under dark condition at 4 °C. The mixed working standard solution was prepared by diluting the mixed stock solution with methanol.

2.4. HPLC-UV conditions

The mobile phase consisted of acetonitrile (A) and 0.8% formic acid aqueous solution (B). The gradient elution was carried out, starting from 10% to 24% A in 8 min, held for 2 min, then to 40% A in 3 min and held for 5 min. The flow rate of the mobile phase was 0.5 mL min^{-1}. The column temperature of 25 °C was maintained. The injection volume of analytical solution was 20 μL. The detection wavelength was 270 nm for TC and OTC and 275 nm for CAP.

2.5. Samples

Honey samples, including the jujube blossom honey (Sample 1), lime tree honey (Sample 2 and 3), motherwort honey (Sample 4) and snow honey (Sample 5 and 6), were purchased from a local market. Except for the experiments mentioned in Section 3.3, which were performed with Samples 1–5, all other experiments were performed with Sample 6.

The freshly-spiked samples containing TC, OTC and CAP were prepared by spiking the mixed working standard solutions into

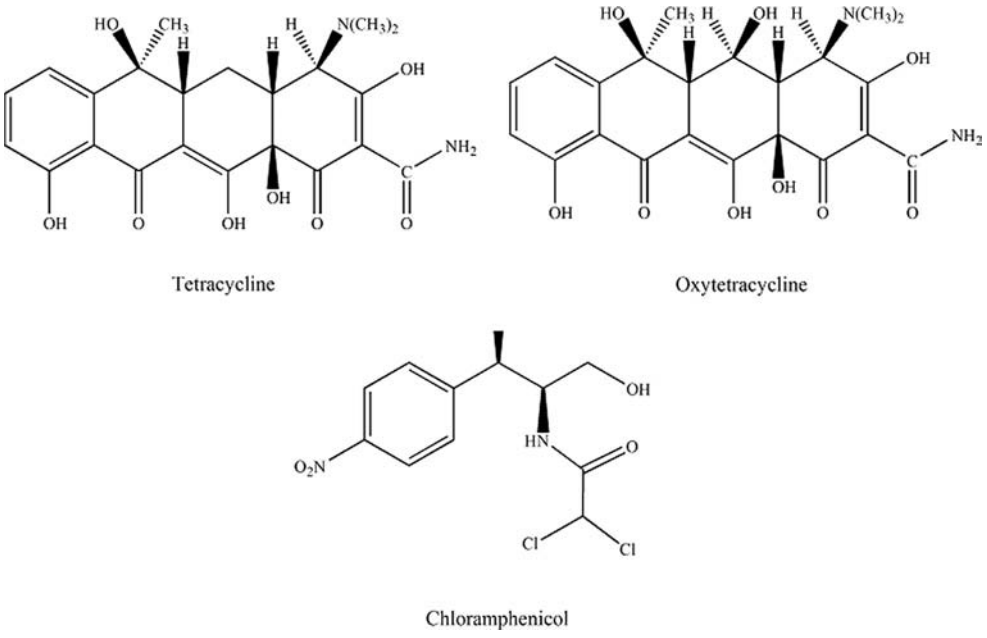

Fig. 2. Chemical structures of TC, OTC and CAP.

honey samples and shaking for 10 min. The aged spiked samples were prepared by the same method except that the spiked samples were kept in sealed bottles for 0, 1, 2, 3, 4, 5, 7, 9 weeks, respectively.

2.6. Aqueous two-phase extraction

2.0 g of honey sample was mixed with 4 mL of 0.1 mol L^{-1} EDTA solution in 10 mL centrifuge tube. 0.1154 g of SDS was accurately weighed and added in the solution. 169 μL of O[MIM]Br and 0.08 g of NaCl were added into the tube. The pH of the mixture was adjusted to 5.0 with hydrochloric acid. The mixture was ultrasonically shaken for 1 min and centrifuged for 3 min at 7000 rpm. The ATP was formed and analytes were extracted into the upper phase. 100 μL of the upper phase was diluted with 25 μL acetonitrile. The resulting solution was filtered with 0.22 μm PTFE filter membrane before analysis.

3. Results and discussion

3.1. Optimization of extraction conditions

In order to obtain high extraction efficiency, the effects of experimental parameters, including the volume of ionic liquid, the category and amount of salts, sample pH value, extraction time and temperature were investigated.

3.1.1. Volume of Ionic liquid

The volume of ionic liquid directly affects the formation of ATP and the extraction of analytes. The concentration of SDS was fixed at 0.1 mol L^{-1}. With the increase of ionic liquid concentration, a homogeneous solution with low-viscosity was first observed and then tiny drops dispersing in the solution could be found, which meant that a SDS-rich ATP was formed. When the molar ratio of ionic liquid to SDS increased to nearly 1:1, precipitation occurred. With further increase of ionic liquid concentration, the precipitate dissolved and tiny drops occurred again, which meant that a IL-rich ATP was formed. With the further increase of ionic liquid concentration, a low-viscosity homogeneous solution was formed again. Compared with the SDS-rich ATP, the IL-rich ATP was more stable and a clearer phase interface was formed. Therefore, the IL-rich ATP was chosen as the extraction system. A series of experiments were performed by adding different volume of ionic liquid (165–173 μL). The recoveries of the analytes first slightly increase and then slightly decrease with the increase of volume of ionic liquid, and are highest when the volume is 169 μL corresponding to 0.18 mol L^{-1} in the system. 169 μL was selected as the volume of ionic liquid.

3.1.2. The category and amount of salts

Addition of inorganic salt may cause a modification of both intermicellar and intramicellar interaction. It is shown that when cationic and anionic surfactants are not equimolar, salts effect is mainly dependent on the oppositely charged counterion. The effects are attributed to the reduced repulsion between the surfactant headgroups, induced by binding of the counterions on the micellar headgroups [25,26]. In the research, some salts including potassium carbonate (K_2CO_3), ammonium sulfate ($(NH_4)_2SO_4$), dipotassium hydrogen phosphate (K_2HPO_4) and NaCl were used. It was found that inorganic salts can help in forming the ATP. When NaCl was added in the system, a clearer and more stable interface was formed. Therefore, NaCl was chosen. In the presence of NaCl, phase separation was very fast owing to the increase in density difference between the two phases. The effect of NaCl amount in the range of 0.03–0.3 g was also investigated. With the increase of NaCl amount, the recoveries first increase and then decrease, and the volume of the upper phase increases which can result in the decrease of enrichment factor. When the amount of NaCl was 0.3 g, ATP disappeared and a homogeneous phase was formed. Therefore, 0.08 g was selected as the amount of NaCl.

3.1.3. Selection of pH value of sample solution

The pH value of sample solution can affect the solubility of the analytes. Therefore, the effect of pH value of the sample solution in the range of 2–12 on the extraction recoveries was studied. The results are shown on Fig. 3. The optimal extraction recoveries are obtained at pH 5. The reason is that the target analytes are amphoteric compounds. When the pH value was 2 or lower, precipitation was formed in the system due to the formation of ethylenediaminetetraacetic acid.

3.1.4. Effect of ultrasound extraction time

Aqueous two phase extraction is a type of equilibrium extraction, and the optimal extraction efficiency is obtained once the equilibrium is established. Hence, the effect of ultrasound time on extraction efficiency was investigated in the range of 0–16 min. The experimental results indicated that the recoveries of the analytes were unchanged when the extraction time was longer than 1 min. The extraction equilibrium can be achieved within 1 min and longer extraction time did not affect the extraction efficiency. This was probably because the contact surface area between the analytes and the extraction phase was very large due to the tiny drops of upper extraction phase formed evenly in the solution. The extraction equilibrium can be achieved in short time and the phase transfer of the target analytes was fast.

3.1.5. Extraction temperature

The effect of temperature on the extraction recovery was evaluated from 10 to 50 °C.No obvious difference of recoveries was found, indicating that the system was not sensitive to temperature change.

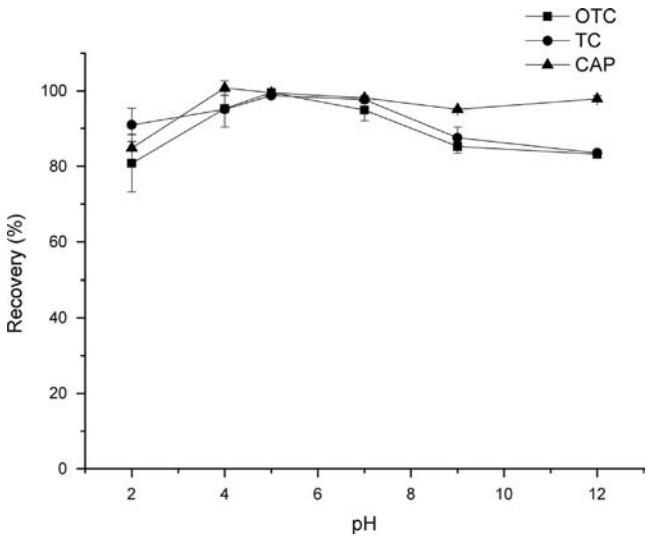

Fig. 3. Effect of pH value of sample solution. IL volume, 169 μL; The amount of NaCl, 0.08 g; Ultrasound extraction time, 1 min. Each data point represents three samples. Error bars represent standard deviations.

3.2. Method validation

The method was validated by parameters, including specificity, limit of detection (LOD), limit of quantitation (LOQ), linear range, precision, accuracy and robustness.

3.2.1. Specificity

TC, OTC and CAP were determined under the optimized conditions. Fig. 4 shows HPLC chromatograms of the honey sample. Typical chromatograms of blank honey sample and spiked honey sample were compared to evaluate the specificity. It is seen from the chromatography of blank honey sample that there are no interfering peaks at the retention times of TC and OTC and the very weak peak at the retention time of CAP should not affect the determination of CAP.

3.2.2. LOD, LOQ and linear range

The working curves were constructed by plotting the peak areas measured versus the concentrations of analytes in the spiked samples. The slope and intercept of the linear regression equations, the residual standard deviations ($S_{y/x}$) and correlation coefficients for the analytes determining are listed in Table 1. LODs and LOQs indicated in Table 1 are determined as the lowest concentrations yielding a signal-to-noise (S/N) ratio of 3 and 10, respectively. The MRLs of the target analytes in the samples are higher than the LOQs. So the LOQs and linear regression equations are appropriate to the goal of the present method.

3.2.3. Precision

Precision was evaluated by measuring intra- and inter-day relative standard deviations (RSDs). The intra-day precision was obtained by analyzing spiked samples five times in one day. The inter-day precision was obtained by analyzing spiked samples over

Table 1
Regression equations, LODs and LOQs.

Analyte	Regression equation ($n=5$)	Correlation coefficient	linear range (μg/kg)	LOD (μg/kg)	LOQ (μg/kg)
OTC	$A=(0.30\pm0.004^{a})$ $C+(-1.06\pm1.372^{b})$	0.9997	30.3–303.6	8.2	27.4
TC	$A=(0.25\pm0.002^{a})$ $C+(0.56\pm0.356^{b})$	0.9998	20.1–301.2	5.8	19.5
CAP	$A=(0.66\pm0.005^{a})$ $C+(-1.38\pm0.909^{b})$	0.9999	20.4–305.4	4.2	13.8

[a] Standard deviation of slope.
[b] Standard deviation of intercept.

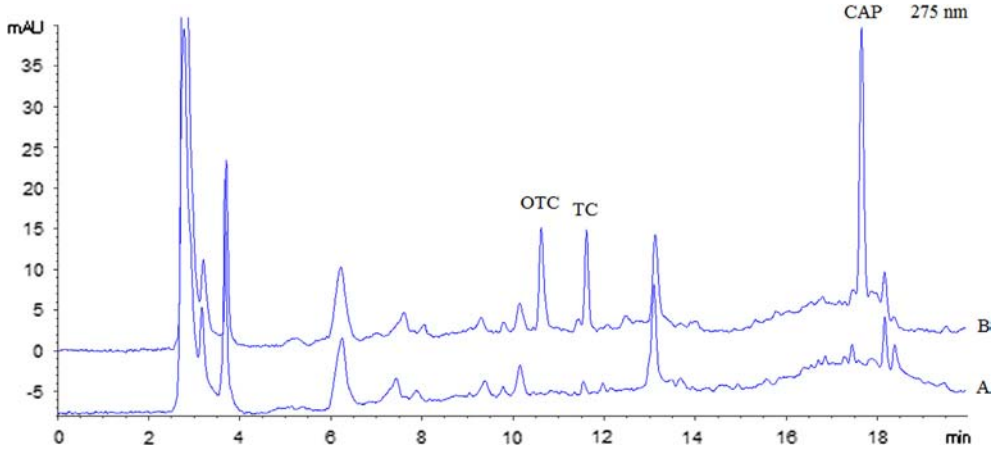

Fig. 4. Chromatograms of Sample 6 (A), spiked Sample 6 (B).

Table 2
Intra-day and inter-day precision at low, medium and high concentration of analyte.

Analyte	Intra-day precision (RSD, %, $n=5$)			Inter-day precision (RSD, %, $n=5$)		
	Low (40 μg/kg)	Medium (100 μg/kg)	High (200 μg/kg)	Low (40 μg/kg)	Medium (100 μg/kg)	High (200 μg/kg)
OTC	4.6	1.8	2.0	6.7	6.3	5.5
TC	3.9	3.2	2.4	4.8	4.5	4.0
CAP	4.8	2.8	2.7	5.9	4.6	4.7

Table 3
Analytical results of honey samples ($n=5$).

Sample	Added (μg/kg)	OTC		TC		CAP	
		Recovery (%)	RSD (%, $n=5$)	Recovery (%)	RSD (%, $n=5$)	Recovery (%)	RSD (%, $n=5$)
Sample 1	40.0	101.7	3.5	96.4	5.2	108.0	4.4
	100.0	108.4	3.7	99.5	4.7	104.7	5.7
	200.0	99.5	1.4	108.3	3.0	100.5	2.6
Sample 2	40.0	108.4	4.1	90.6	1.7	92.7	5.7
	100.0	103.4	2.7	85.5	4.8	103.9	6.9
	200.0	97.5	1.4	88.8	1.6	108.1	4.3
Sample 3	40.0	100.9	2.4	86.5	1.7	89.5	6.6
	100.0	109.0	0.8	94.8	4.6	101.6	6.3
	200.0	97.3	5.0	88.5	5.0	102.6	6.6
Sample 4	40.0	91.6	4.4	109.4	6.1	104.2	2.1
	100.0	92.8	1.8	101.6	1.5	103.9	1.8
	200.0	103.2	1.4	108.2	0.7	106.7	2.5
Sample 5	40.0	109.6	0.9	97.2	5.8	110.9	2.4
	100.0	104.3	2.4	91.1	1.3	94.4	4.7
	200.0	103.1	2.3	94.3	3.9	110.4	3.4

Table 4
Experimental design and recovery measurement.

Factor			Recovery (%)					
IL (μL)	NaCl (g)	pH	OTC		TC		CAP	
			a	*b*	*a*	*b*	*a*	*b*
169	0.08	5	98.8	96.8	98.4	99.2	99.3	98.7
169	0.075	6	93.0	94.6	97.6	96.1	96.1	94.7
170	0.08	6	97.7	95.5	95.2	93.0	96.8	95.8
170	0.075	5	92.5	95.0	94.0	96.2	94.9	96.5

a: first data set
b: second data set

five days. The results are presented in Table 2 and indicate that the present method has good precision.

3.2.4. Accuracy

Samples 1–5 were analyzed to evaluate the accuracy and applicability of the present method. The analytes in the samples were undetectable. The spiked samples were analyzed and the results are shown in Table 3. Recoveries are between 85.5% and 110.9%. The accuracy is acceptable. Therefore, the present method is suitable for the analysis of honey samples.

3.2.5. Robustness

A Plackett–Burman design (3 factors and 2 levels, $N=4$) for the evaluation of robustness effects was applied. The three factors are the volume of IL, the amount of NaCl and the pH value of sample solution respectively. The experiment was carried out in two replicates and the obtained recoveries of the analytes are listed in Table 4. T-test was used to evaluate robustness. The equation for calculating t is listed below [27]:

$$t = \frac{\text{average effect}}{2\sqrt{\left[\sum d^2/(N/8)\right](N/8)/\sqrt{2N}}}$$

T-test results are given in Table 5. Average recoveries are calculated based on the results given in Table 4. Effect of each factor is the difference between recoveries. d represents the difference between effects and N represents the experimental number. All the t-values are less than 3.18, the 5% critical t-value associated with 3 degree of freedom. Based on the statistical analysis, the robustness of the method should be acceptable.

3.3. Stability of TC, OTC and CAP in honey

The spiked samples containing TC, OTC and CAP were prepared and stored in a clear plastic container at ambient temperature. The samples were exposed to indirect sun light for 0, 1, 2, 3, 4, 5, 7, 9 weeks, respectively. The samples were analyzed by the present method and the results are shown on Fig. 5. The concentrations of TC and OTC decrease and the concentration of CAP is unchanged with the increase of the storage time. After 9 weeks of storage, the recoveries of TC, OTC and CAP are 66.3%, 59.6% and 97.9%, respectively.

4. Conclusion

TC, OTC and CAP in honey were extracted by IL/AS-based ATPE. The parameters affecting the extraction efficiency, including the volume of ionic liquid, the category and amount of salts, sample pH value, extraction time and temperature were investigated. IL/AS-based ATPE is an easy and rapid pretreatment method of honey samples. The present method is environmentally friendly because of the avoidance of volatile solvents. Compared with traditional IL/salt ATPE, the amount of ionic liquid and salts used to form ATPs is

Table 5
Arithmetric treatment of recovery data.

Factor	Level	OTC						TC						CAP					
		1st data set		2nd data set		Average effect	d	t	1st data set		2nd data set		Average Effect	d	t	1st data set		2nd data set		Average Effect	d	t
		Average Recovery (%)	Effect	Average recovery (%)	Effect				Average recovery (%)	Effect	Average recovery (%)	Effect				Average recovery (%)	Effect	Average recovery (%)	Effect			
IL (μL)	169	95.9		95.7					98.0		97.6					97.7		96.7				
	170	95.1	+0.8	95.2	+0.5	+0.65	+0.3	0.55	94.6	+3.4	94.6	+3.0	+3.2	+0.4	3.16	95.8	+1.9	96.2	+0.5	+1.2	+1.4	1.75
NaCl (g)	0.08	98.2		96.2					96.8		96.1					98.0		97.2				
	0.075	92.8	+5.4	94.8	+1.4	+3.4	+4.0	2.89	95.8	+1.0	96.2	−0.1	+0.45	+1.1	0.45	95.5	+2.5	95.6	+1.6	+2.05	+0.9	2.98
pH	5	95.6		95.9					96.2		97.7					97.1		97.6				
	6	95.4	+0.2	95.0	+0.9	+0.55	−0.7	0.47	96.4	−0.2	94.6	+3.1	+1.45	−3.3	1.43	96.4	+0.7	95.2	+2.4	+1.55	−1.7	2.26

d:Difference between effects
t:*t*-values of *t*-test

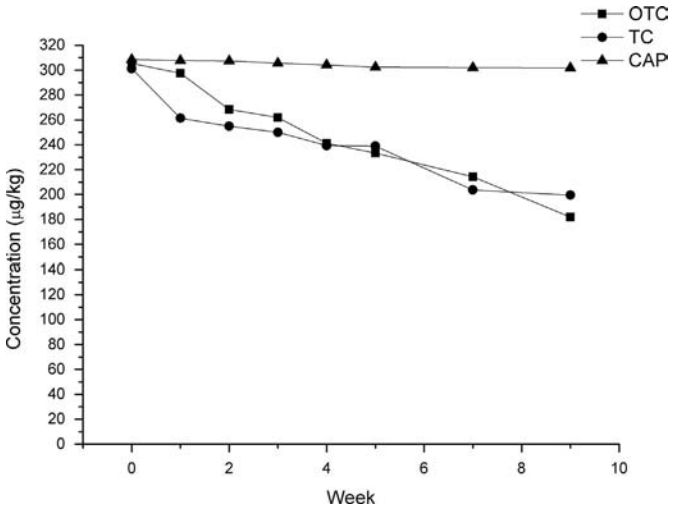

Fig. 5. Stability of TC and OTC in honey samples. Concentrations of both TC and OTC was 300 μg kg^{-1}.

smaller and the phase separation time is shorter. It seems possible to extend this method to the extraction of antibiotics in other similar fluid samples by varying the extraction conditions.

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