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Selective extraction and separation of oxymatrine from *Sophora flavescens* Ait. extract by silica-confined ionic liquid

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ABSTRACT

This study highlighted the application of a two-stepped extraction method for extraction and separation of oxymatrine from *Sophora flavescens* Ait. extract by utilizing silica-confined ionic liquids as sorbent. The optimized silica-confined ionic liquid was firstly mixed with plant extract to adsorb oxymatrine. Simultaneously, some interference, such as matrine, was removed. The obtained suspension was then added to a cartridge for solid phase extraction. Through these two steps, target compound was adequately separated from interferences with 93.4% recovery. In comparison with traditional solid phase extraction, this method accelerates loading and reduces the use of organic solvents during washing. Moreover, the optimization of loading volume was simplified as optimization of solid/liquid ratio.

1. Introduction

Alkaloid is a kind of organic compound having alkaline properties and containing nitrogen, which can be extracted from plant (e.g. potato, tomato, coffee bean) or animal (e.g. shellfish, puffer fish) foods for treatment of certain diseases [1]. To obtain alkaloids for bioassays or pharmaceutical production, they are extracted from raw materials with subsequent analysis and separation. Comparing with extraction, latter processes are more complex and important. Thus, much work has attempted to improve the analysis and separation of alkaloids from extracts, with chromatography being considered effective and adopted as the prevalent method [2–5]. Stationary phases or adsorbents are important in chromatography and related techniques (e.g. solid phase extraction), which significantly affect the process and efficiency. Therefore, improvement of these materials' performances continuously attracts the attention of scientists.

Previous works have reported silica-confined ionic liquids (SiILs) being used in high-performance liquid chromatography (HPLC) [6,7] and solid phase extraction (SPE) [8,9] due to their excellent physical and chemical properties. The multi-interactions between alkaloids and ionic liquids (ILs) have already been investigated in work related to the application of ILs for extraction [10,11]. In this case, SiILs show potential applicability as an alternative stationary phase or adsorbent for the improved separation of alkaloids when compared with conventional materials (e.g. C_{18}, amino-silica). To test this, several SiILs were employed to analyze and separate oxymatrine from *Sophora flavescens* Ait. (SFA) extract.

S. flavescens Ait., a functional food or nutraceutical, contains many alkaloids, with matrine and oxymatrine being the most abundant. Although their structures are similar, the latter is preferred due to its excellent clinical efficiency [12–15]. Methods such as chromatography [16,17], SPE [18] and adsorption [19] have been tested for the analysis and separation of oxymatrine from extract, found to be complicated and inefficient. To overcome these, an efficient method should be proposed with relative simple process.

Traditional SPE generally involves loading, washing, and eluting with loading being the most time-consuming part of the process, because it requires time to achieve adequate adsorption of the target compounds onto sorbent in the presence of interfering species. To accelerate loading and to reduce the use of organic solvents during washing, sorbents can be directly mixed with extract; target compounds then quickly adsorb, assisted by shaking, without excessive interference. Moreover, the optimization of loading volume can be simplified as optimization of solid/liquid ratio. This work reports the extraction of oxymatrine from SFA extract to SiILs by adsorption with subsequent SPE (Fig. 1). The method was simple, adaptable and efficient. Target compound was adsorbed on the sorbent without much interference, which decreased the requirement of washing solvents. All necessary conditions were systematically optimized through experimentation, and the interactions between SiILs and the target compounds were investigated.

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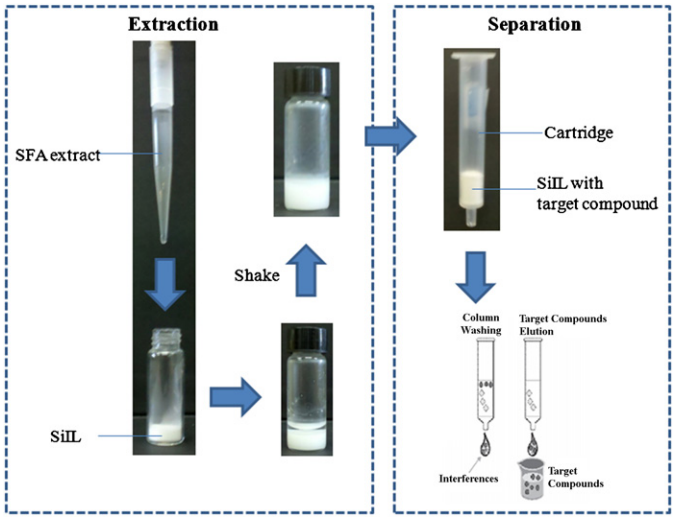


Fig. 1. Scheme of extraction and separation of oxymatrine from SFA extract.

2. Experimental

2.1. Chemicals

(3-Chloropropyl)trimethoxysilane (97%), (3-aminopropyl) trimethoxysilane (97%), bis(trifluoromethane)sulfonimide lithium salt (99%), sodium tetrafluoroborate (98%) and hexafluorophosphoric acid solution (*ca.* ~65 wt.% in water) were purchased from Sigma (St. Louis, MO, USA). Imidazole (99%) and 1-methylimidazole (99%) were obtained from Aldrich (Milwaukee, WI, USA). 1-Ethylimidazole (>98%) and 1-butylimidazole (>98%) were purchased from Tokyo Chemical Industry Co. Ltd. (Tokyo, Japan). Matrine and oxymatrine were purchased from the National Institute for the Control of Pharmaceutical and Biological Products (Beijing, China). Acetonitrile, ethanol, methanol, *n*-hexane and methylene chloride were obtained from Pure Chemical Co., Ltd. (Ansan, Korea). Distilled water was filtered using a vacuum pump (Division of Millipore, USA) and a filter (HA-0.45, Division of Millipore, USA) before use. The 15.0 μm Apex silica and RP-18 particles were purchased from Merck Chemicals Ltd. All other solvents used in the experiment were LC or analytical grade. All the samples were filtered by using a filter (MFS-25, 0.2 μm TF, WHATMAN, USA) before injection into the HPLC system.

2.2. HPLC analysis

The chromatography system consisted of Waters 600 s Multi solvent Delivery System, Waters 616 liquid chromatography (Waters Associates, Milford, MA, USA), a Rheodyne injector (20 μL sample loop) and a variable wavelength 2487 UV dual channel detector. Data processing was carried out with a Millenium 3.2 consisted of HP Vectra 500PC. The HPLC analysis was performed with a commercial C_{18} column (4.6 mm × 150 mm, 5.0 μm) from RStech (Daejeon, Korea). The mobile phase was methanol/water/trifluoroacetic acid (16:84:0.002, *v/v/v*) with the flow rate of 0.5 mL min^{-1}, and the UV wavelength was at 220.0 nm [18].

2.3. Preparation of SilLs

The experimental procedure has been described in the literature [20]. Silica was first immersed in hydrochloric acid for 24.0 h and then washed with deionized water and dried at 100 °C for 8.0 h. The activated silica (5.0 g) was suspended in 50.0 mL dry toluene, after which an excess of 3-chloropropyltrimethoxysilane (5.0 mL) was added. After the suspension was refluxed for 12.0 h, the reaction was stopped after the system was cooled to room temperature. The particles were washed in turn with toluene, deionized water, and methanol. The chloropropyl silica, SilprCl, was dried at 60 °C for 10.0 h.

The chemically bonded chloropropyl groups on the silica surface were subsequently reacted with imidazole, 1-methylimidazole, 1-ethylmethylimidazole and 1-butylimidazole. In brief, 5.0 g dry chloropropyl silica was added to 50.0 mL anhydrous toluene and a large excess of imidazoles (5.0 g). The mixture was refluxed with stirring for 18.0 h. The cooled modified silica was washed in turn with toluene, ethanol, and methanol. The silica chemically bonded with imidazolium was dried at 60.0 °C for 10.0 h. The silica bonded with imidazole, 1-methylimidazole, 1-ethylmethylimidazole and 1-butylimidazole were labeled SilprImCl, SilprMImCl, SilprEImCl and SilprBImCl, respectively.

Aminopropyl silica ($SilprNH_2$) was obtained by the reaction of 3-aminopropyltrimethoxysilane (5.0 mL) with activated silica (5.0 g) using a procedure as that used in the preparation of SilprCl.

According to Ref. [21], the SilprImCl was ion exchanged with $Li(Tf_2N)$ and HPF_6 to obtain $SilprImTf_2N$ and $SilprImPF_6$, respectively. Into a 200.0 mL flask was added 5.0 g SilprImCl and an excess of $Li(Tf_2N)$ and HPF_6 was added, followed by 50.0 mL deionized water. After 12.0 h of stirring, $SilprImTf_2N$ and $SilprPF_6$ were obtained and washed sequentially with ethanol and methanol. The particles were dried at 60 °C for 10.0 h.

As described in Ref. [22], 3.0 g $NaBF_4$ was added to 5.0 g of SilprImCl in dichloromethane and stirred for 12.0 h to obtain $SilprImBF_4$. The suspension was filtered and washed with water and ethanol, and then the particles were dried at 60 °C for 10.0 h. The chemical structures of the studied stationary phases were listed in Table 1.

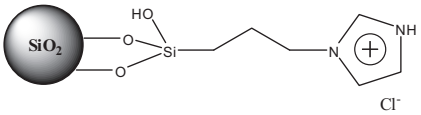
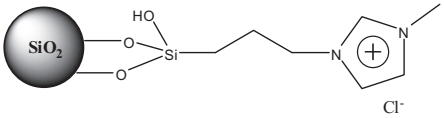
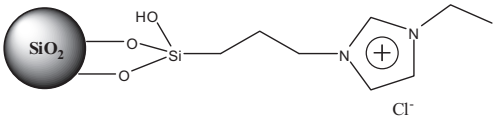
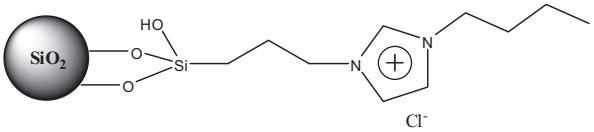
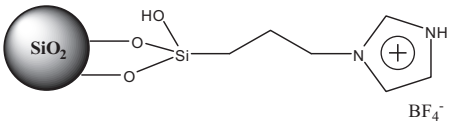
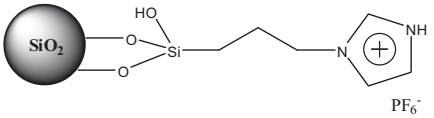
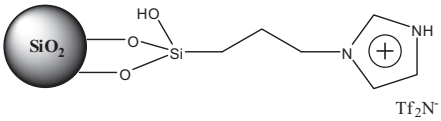
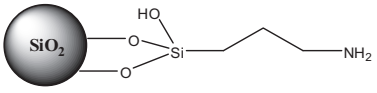
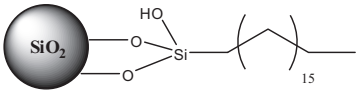
2.4. Extraction

The SilLs were assessed by comparing the amounts adsorbed on different adsorbents by a static method. Into several flasks were placed 400.0 mg particles, and 1.0 mL of standards of matrine and oxymatrine at a concentration of 0.025 mg mL^{-1} was added, respectively. After the mixture was shaken at 25.0 °C for 30.0 min, the supernatant was collected and filtered (0.2 μm). The concentrations of unadsorbed matrine and oxymatrine in the solution were determined by HPLC with a commercial C_{18} column at room temperature. The adsorbed amounts on the particles were calculated by subtracting the amounts of unadsorbed target compounds from the initial amounts of these compounds. Then the root of SFA samples obtained from local market were ovendried, sliced and crushed, after which 0.5 g of the powdered particles was extracted using 50.0 mL of water for 4.0 h [23]. Similarly, oxymatrine was extracted to SilLs from the obtained SFA extract by adsorption. Repeatability was tested by extracting 3.0 times over a 5-day period. A 2-sided *t*-test was used to evaluate the data of independent samples.

2.5. Separation

The suspensions (1.0 mL) obtained during extraction were added to empty polypropylene cartridges, and then washed and eluted by different solvents: water, methanol, acetonitrile, *n*-hexane and methylene chloride. The filtrates were evaporated to dryness and reconstituted in 1.0 mL mobile phase for further HPLC analysis.

Table 1
Chemical structures and element analysis of studied adsorbents.

No.	Name	Structure	C (%)	H (%)	N (%)	S (%)	Coverage (μmol m^{-2})
1	SilprImCl		11.81	1.95	4.02	–	3.23
2	SilprMImCl		12.34	1.98	3.64	–	2.93
3	SilprEImCl		12.49	1.99	3.24	–	2.60
4	SilprBImCl		12.33	1.97	2.06	–	1.63
5	SilprImBF$_4$		10.96	1.57	3.61	–	2.85
6	SilprImPF$_6$		8.21	1.13	2.51	–	1.88
7	SilprImTf$_2$N		12.05	1.67	4.02	1.55	1.11
8	SilprNH$_2$		8.96	2.19	3.19	–	4.92
9	C$_{18}$		16.76	2.72	–	–	1.78

3. Results and discussion

3.1. Elemental analysis

The carbon, hydrogen, nitrogen and sulfur contents of the intermediates and final materials were determined by elemental analysis that performed on an EA1112 (Italy). The elemental contents and surface coverage of SilprCl, SilprImCl, SilprMImCl, SilprEImCl, SilprBImCl, SilprImBF$_4$, SilprImTf$_2$N, SilprImPF$_6$, SilprNH$_2$ and C$_{18}$ are listed in Table 1. According to Ref. [7], bonding density was 1.78 μmol m^{-2} in C$_{18}$, based on the percentage amounts of carbon. Based on the nitrogen percentages, bonding densities were 3.23, 2.93, 2.60, 1.63, 2.85, 1.88 and 4.92 μmol m^{-2} for SilprImCl, SilprMImCl, SilprEImCl, SilprBImCl, SilprImBF$_4$, SilprImPF$_6$ and SilprNH$_2$, respectively. The surface coverage of SilprImTf$_2$N was 1.11 μmol m^{-2} based on the presence of sulfur. Elemental analysis showed that immobilization on the surfaces was successful. The bonding density of imidazolium silica decreased with increasing alkyl branches of imidazoles. This may be rationalized by steric hindrance from the alkyl chain. However, the separation of analytes was dominated by the influence of interactions between the analytes and the stationary phase, rather than the influence of bonding density [20].

3.2. Extraction

Extraction was achieved by the adsorption of oxymatrine on SiILs. Thus, adsorbed amounts are indicative of the performance of each material. Because matrine is structurally similar to oxymatrine, the former was also investigated.

3.2.1. Types of SiILs

IL's structure significantly influenced its physicochemical properties, affecting the extraction efficiency of the target compound. To find the most suitable SiILs and evaluate their performance, SiILs with different cations and anions were investigated by a static method with 0.025 mg mL^{-1} standard solutions. Featuring the same Cl$^-$ anion, SilprImCl, SilprMImCl, SilprEImCl and SilprBImCl

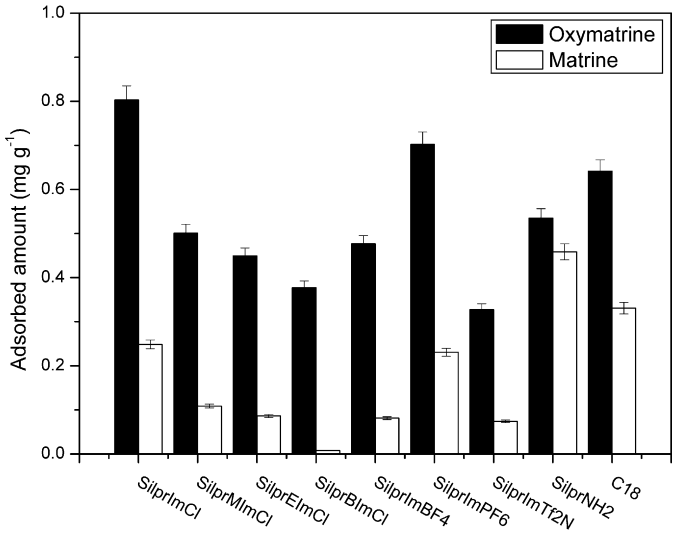


Fig. 2. Effect of SilLs on adsorbed amounts of matrine and oxymatrine (temperature = 25 °C, solid/liquid ratio = 20/1 (mg mL^{-1}), time = 30.0 min).

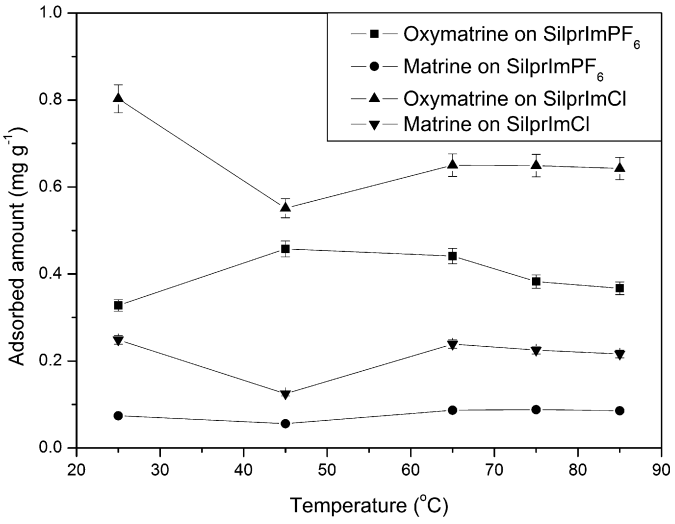


Fig. 3. Effect of temperature on adsorbed amounts of matrine and oxymatrine (solid/liquid ratio = 20/1 (mg mL^{-1}), time = 30.0 min).

were used to investigate the effects of the cations (Fig. 2). By comparing the four adsorbents, the adsorbed amounts decreased with increasing alkyl length in the ILs, with SilprImCl exhibiting the highest adsorption capacity. Although hydrogen bond acidity and hydrophobic interaction increased in series from SilprImCl to SilprBImCl [24], increased hydrophobicity and steric hindrance prevented the SilLs interacting with aqueous matrine and oxymatrine.

The anions also influenced the properties of the ILs. Therefore, stationary phases of SilLs with four different anions (Cl^-, BF_4^-, Tf_2N^- and PF_6^-) were studied (Fig. 2). In comparison with SilprImBF$_4$, SilprImPF$_6$ and SilprImTf$_2$N, SilprImCl had higher adsorbed amounts of matrine and oxymatrine, attributable to the excellent water miscibility of its functional groups. Also, the pH value of SilprImCl was relatively low when it ionized in water [25], possibly aiding adsorption.

The adsorption of matrine and oxymatrine on conventional adsorbents, C_{18} and SilprNH$_2$, were measured for comparison. The adsorption capacity of SilprImCl was the highest due to the multiple interactions (π–π interaction, ionic/charge–charge and dipolar interactions) between sorbent and target compound, and the quite different adsorbed amounts of matrine and oxymatrine on SilprImCl demonstrate its ability to separate these two compounds [26].

3.2.2. Effects of temperature

To investigate the effects of temperature on the adsorption amounts of alkaloids on SilprImCl, adsorption between 25.0 and 85.0 °C was studied. In Fig. 3, the adsorbed amounts of matrine and oxymatrine initially decreased with increasing temperature, but then increased as the temperature increased above 65.0 °C. This may be related to the chemical interactions and physical adsorption between the analyte and the SilLs. The desorption of these compounds have been reported to be expedited with increasing temperature, therefore the adsorbed amounts decreased with increasing temperature up to 45.0 °C. However, diffusion increased with further increases temperature, increasing the amounts adsorbed on SilprImCl. By thoroughly consideration, room temperature (25 °C) was preferred. SilprImPF$_6$ was also examined because its hydrophobicity decreases with increasing temperature [27]. The results show that the adsorbed amounts increased at certain temperature, but remained lower than SilprImCl. Hence, SilprImCl at 25 °C was used in subsequent experiments.

3.2.3. Effects of time

Extractions were carried out with a solid/liquid ratio of 400/1 (mg mL^{-1}) at 25.0 °C for various durations from 15.0 to 35.0 min to optimize the extraction time. The extraction efficiency (EE) was calculated by the following equation:

$$\text{EE}\ (\%) = \left(1 - \frac{C}{C_0}\right) \times 100$$

where C (mg mL^{-1}) is the concentration of target compounds in the extract after extraction and C_0 (mg mL^{-1}) is the initial concentration of the target compounds in the extract. Fig. 4 shows the extracted amounts of oxymatrine dramatically increased as the extraction time increased from 15.0 to 30.0 min. After 30.0 min, no obvious increase in EE was observed.

3.2.4. Effects of solid/liquid ratio

Based on the above optimization, SilprImCl was used to extract oxymatrine from SFA extract. The concentrations of matrine and oxymatrine in the extract were 0.0091 and 0.165 mg mL^{-1}. To optimize the efficiency of oxymatrine extraction, the solid/liquid ratio was studied. Fig. 5 shows that EE increased with increasing amount

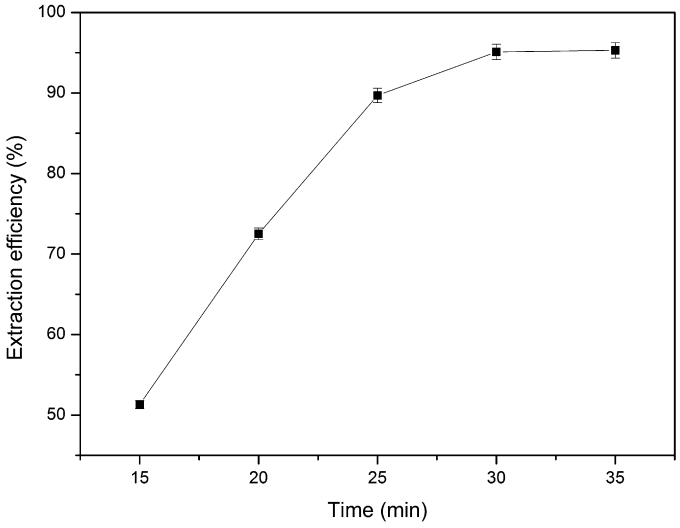


Fig. 4. Effect of time on extraction efficiency of oxymatrine (SilprImCl, temperature = 25 °C, solid/liquid ratio = 400/1 (mg mL^{-1})).

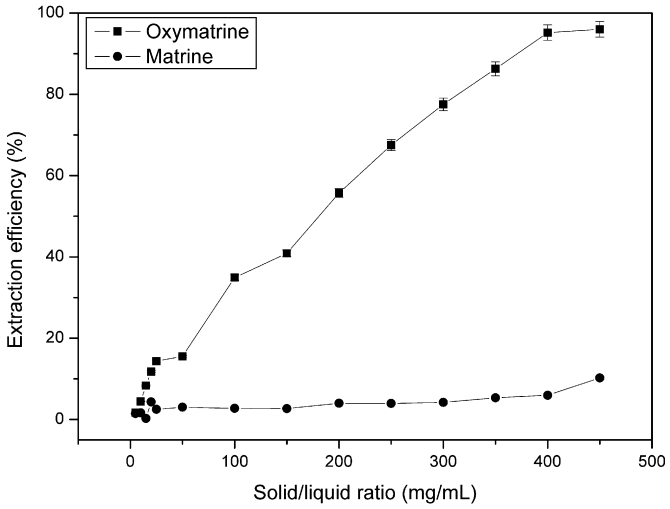


Fig. 5. Effect of solid/liquid ratio on extraction efficiency of matrine and oxymatrine (SilprImCl, temperature = 25 °C, time = 30.0 min).

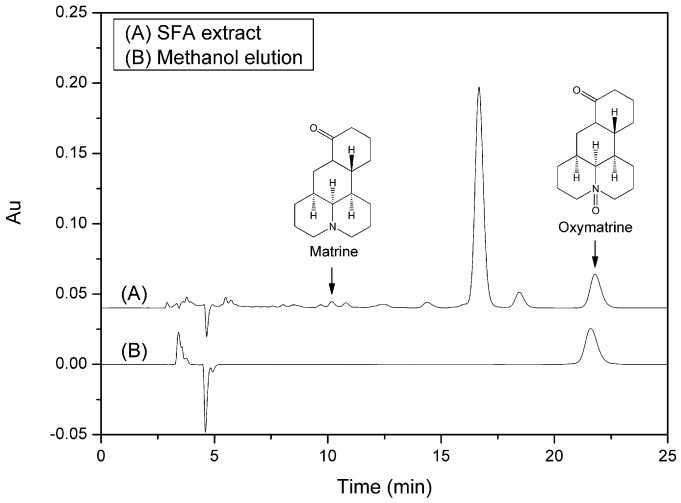


Fig. 6. Chromatogram of SFA extract (A) and methanol elution (B).

Table 2
Eluted amount and recovery of oxymatrine from SFA extract (base on 1.0 mL SFA extract).

Solvent (1.0 mL)	Eluted amount (mg)	Recovery (%)	
		Separation step	Extraction and separation step
Water	–	–	–
n-Hexane	–	–	–
Methylene chloride	–	–	–
Methanol	15.37	98.2	93.4
Ethanol	7.52	48.1	45.7
Acetonitrile	12.52	80.1	76.1

of SilprImCl. Given economic considerations, 95.1% EE of oxymatrine was considered optimal when a solid/liquid ratio of 400/1 (mg mL^{-1}) was used. The extraction of bioactive components to the solid phase is a physical process. With increasing amounts of SilprImCl, the chance of the bioactive molecules coming into contact with SilprImCl increased, leading to higher EE.

Another phenomenon was also observed in the experiment. The EE of matrine is found to be much lower than that of oxymatrine, which may attribute to competitive adsorption. According to Ref. [28], some experiments were performed to examine the competitive adsorption by comparing the adsorption behaviors of matrine and oxymatrine in binary (mixture of matrine and oxymatrine) and single (matrine or oxymatrine only) system. At the concentrations of 0.025 mg mL^{-1} and 0.05 mg mL^{-1}, it was found that the adsorption of oxymatrine (0.80 mg g^{-1}, 1.71 mg g^{-1}) increased in binary system compared with the oxymatrine only system (0.68 mg g^{-1}, 1.02 mg g^{-1}). Conversely, in binary systems, adsorption of matrine (0.25 mg g^{-1}, 0.48 mg g^{-1}) decreased compared with the matrine only system (0.28 mg g^{-1}, 0.53 mg g^{-1}). Hence, most of the matrine remained in the extract, and thus the oxymatrine and matrine were separated from each other.

3.3. Separation

The suspension obtained during extraction was added to cartridges for SPE process. SPE allowed the separation of oxymatrine from interferences. As shown in Table 2, water, *n*-hexane and methylene chloride could not wash the target compound from SilprImCl. Therefore non-toxic water was selected as the washing solvent for the removal of interferences. Most of the oxymatrine could be eluted from the SilprImCl sorbent by methanol (0.153 mg mL^{-1} extract), acetonitrile (0.125 mg mL^{-1} extract) or ethanol (0.075 mg mL^{-1} extract). Methanol was selected as the elution solvent due to its higher recovery. The volumes of each solvent were optimized through the testing of different volumes (from 1.0 to 4.0 mL). When the washing solvent was used at volumes greater than 1.0 mL, the removal of interferences did not increase. The eluted amount of oxymatrine (0.153 mg based on 1.0 mL SFA extract) did not increase when the elution solvent was used at volumes larger than 1.0 mL. Therefore, 1.0 mL washing water and 1.0 mL eluting methanol were considered optimal. The chromatogram is shown in Fig. 6. The SilprImCl could be regenerated by washing with triethylamine/methanol (5/95, *v/v*) and drying at 60.0 °C.

3.4. Recycling of SilprImCl

The recycling of SilprImCl for extraction and separation of oxymatrine from SFA extract was investigated. The recovery yields of oxymatrine by SilprImCl over four cycles from SFA extract progressed from 93.4 to 89.7%. The recovery yields of oxymatrine exhibited little decrease, indicating that this sorbent is stable.

3.5. Analytical performance

A series of experiments were undertaken to examine the extraction method's linearity, precision, detection limits and other characteristics of the method under the optimized conditions. Oxymatrine had good linearity from 5.0×10^{-3} to 0.5 mg mL^{-1}, with correlation coefficients (r) of 0.9998. Precision was determined by the six times repeated analysis of the samples after extraction and separation with a standard solution of a single concentration, and the RSD was 2.1%. Based on a signal-to-noise ratio of 3, the limits of determinations (LODs) of oxymatrine were 53.2 ng mL^{-1}. These results show that the proposed method is stable, with wide potential applicability to the determination of other alkaloids.

4. Conclusion

The proposed material and method were successfully applied to extraction and separation of oxymatrine from SFA extract. First, 95.1% oxymatrine was extracted to SilprImCl from SFA extract with a solid/liquid ratio of 400/1 (mg mL^{-1}) at 25.0 °C in 30.0 min. Meanwhile, oxymatrine was separated from matrine. A SPE process was then involved to separate oxymatrine from other interferences,

and a total 93.4% oxymatrine (based on 1.0 mL SFA extract) was obtained after washing by 1.0 mL water and eluting by 1.0 mL methanol. The whole process was simple and efficient. Moreover, this method can be developed as an integrated process including extraction, concentration and separation. Due to the high levels of oxymatrine in SFA extract, concentration was not necessary. The reported method exhibited potential applicability to other alkaloids in functional foods, medicinal herbs and nutraceuticals.

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