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Screening, recognition and quantitation of salbutamol residues in ham sausages by molecularly imprinted solid phase extraction coupled with high-performance liquid chromatography–ultraviolet detection

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ABSTRACT

A highly selective molecularly imprinted solid phase extraction (MISPE) coupled with liquid chromatography–ultraviolet detection was developed for the determination of salbutamol (SAL) in ham sausages. New molecularly imprinted polymers (MIPs) were synthesized with phenylephrine as dummy template and it revealed good affinity to SAL in methanol–acetonitrile system. Adsorption capacity of the MIPs was evaluated by dynamic adsorption experiments. The MIPs were used as SPE sorbent for the selective clean-up and pre-concentration of SAL in ham sausage samples. The results showed that the matrix compounds presented in ham sausage samples could be removed effectively and the recoveries of SAL at three spiked levels were ranged from 82.6 to 100.5% with the relative standard deviation (RSD) of less than 3.6%. This method is simple and sensitive, and is therefore an alternative tool to the existing methods for analyzing residual SAL in biological samples.

1. Introduction

Recently, owing to the large numbers of food safety events, among which was “lean meat powder” toxic incident, occurred one after another, the problem of food safety has aroused in the world scope and become the focus of the government and the customers [1]. Salbutamol (SAL) is a selective β_2-adrenoceptor agonist extensively used as a bronchodilator in asthmatic patients [2]. Furthermore, SAL is also used as growth-promoting agent in various animals to increase feeding efficiency and carcass leanness [3]. The residues of SAL, which is most abundant in liver and meat, can be harmful to human beings [4]. To ensure food safety, the maximum residue limits of SAL in various foodstuffs have been established in many countries [2,5,6]. Therefore, monitoring the residues of SAL in meat and other animal products used for human consumption is very important.

In order to analysis of SAL in biological samples, several methods have been developed, such as high performance liquid chromatography (HPLC) [1,7,8], capillary electrophoresis (CE) [9–11], gas chromatography–mass spectrometry (GC–MS) [12], liquid chromatography–mass spectrometry (LC–MS) [13,14], enzyme immunoassay [15], and bromatometric methods [16]. However, they are suffered to achieve separation of target compounds from complex sample matrices directly without the pretreatment process. Consequently, a clean-up step is crucial to improve the sensitivity and the specificity before instrumental analysis. Until now, liquid–liquid extraction [17] and solid-phase extraction (SPE) [18], dispersive liquid–liquid microextraction [19,20], matrix solid-phase dispersion [21–24], supercritical fluid extraction [25,26], ionic liquids extraction [27,28] are still recognized as the most commonly used techniques to extract the analyte from biological and food samples. In which, SPE is frequently used for enrichment and clean-up of biological samples due to its benefits of simplicity, rapidness, and little consumption of organic solvents. Nevertheless, the traditional SPE sorbents (C_8, C_{18}, SCX, PCX, HLB, etc.) are lack of special selectivity which commonly leading co-extraction of impurities from sample matrix. Therefore, the development of selective sorbent material for SPE is desired.

For SPE protocol, amongst the best candidates as sorbents for performing selective extractions are molecularly imprinted polymers (MIPs). MIPs are synthetic sorbents designed to retain the target molecule selectively regardless of the complexity of the matrix it is in. Molecular imprinting technique is based on a process where functional monomer and cross-linker are copolymerized in the presence of template molecules, involving the formation of imprinted cavities in which the template is arranged. After polymerization, the template is removed leaving in the polymer network binding sites with shape, size and chemical functionality complementary to the target analyte. The synthesis process is relatively easy, low-cost and the resulting polymers can exhibit high

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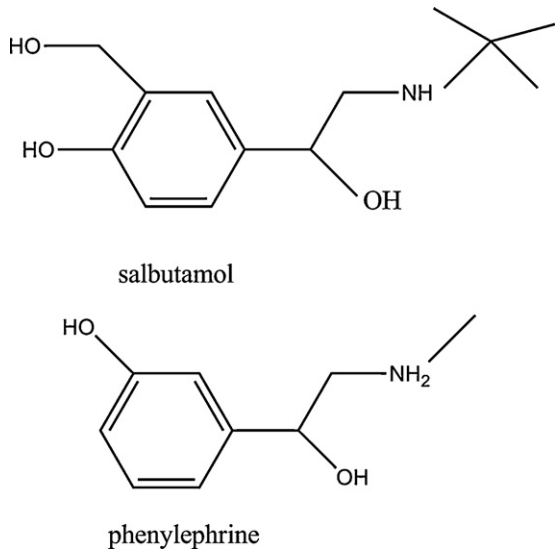


Fig. 1. Molecular structure of salbutamol and phenylephrine.

recognition ability, mechanical and chemical stability and applicability in harsh chemical media [29–32]. Owing to these advantages, MIPs as sorbent material for SPE is potentially one of the most exciting applications for the selective extraction or the clean-up of target analyte from various complex matrices [33,34].

The object of this study was to synthesize MIPs using phenylephrine as dummy template and applied it as special sorbents of SPE to selectively extract SAL in ham sausages. This proposed protocol provided special selectivity of SPE and eliminated the effect of template leakage of MIPs on quantitative analysis. Therefore, it could be potentially applied for the selective separation and quantitative determination of SAL in complicated food samples.

2. Experimental

2.1. Materials

Salbutamol (SAL, Fig. 1) was purchased from the National Institute for the Control of Pharmaceutical and Biological Products (Beijing, China). Phenylephrine, methanol, acetonitrile, acetone, propyl alcohol, chloroform, tetrahydrofuran, ammonia, acetic acid and trifluoroacetic acid (TFA) were obtained from Huaxin Chemical Reagent (Baoding, China). Methacryclic acid (MAA), acrylamide (AM), 4-vinyl pyridine (4-VP), 2-hydroxyethyl methacrylate (2-HEMA) and 2,2-azodiisobutyronitrile (AIBN) were purchased from Kermel Chemical Co. Ltd. (Tianjin, China). Ethylene glycol dimethacrylate (EDMA) was obtained from Sigma–Aldrich (MO, USA). All the other reagents used in the experiment were of the highest grade commercially available. PCX and C_{18} cartridges (3.0 mL, 60 mg) were obtained from Varian Co. (Palo Alto, CA, USA). HLB and SCX cartridges (3.0 mL, 60 mg) were acquired from Sigma (Louis, MO, USA). Double deionized water was filtered through a 0.45 μm fiber membrane before use.

2.2. Instrumentation and conditions

HPLC analysis was performed using a Shimadzu HPLC system equipped with two LC-20AT Solvent Delivery Units, a SUS20A gradient controller, and a SPD-20A UV–VIS Detector (Shimadzu, Kyoto, Japan). An N-2000 workstation (Zheda Zhineng Co. Ltd., Hangzhou, China) was used as the data acquisition system. The analytical columns (250 mm × 4.6 mm I.D., C_{18}, 5 μm) were obtained from YMC Co. Ltd. (Kyoto, Japan). The mobile phase was water–methanol (85:15, v/v, containing 1‰ TFA) with a flow rate of 1.0 mL min^{-1}. The injection volume was 10 μL and the detection wavelength of the detector was set at 203 nm. Ultrasonic cleaner (KQ3200E) was purchased from Kunshan Instrument Co. Ltd. (Jiangsu, China) and set at 40 kHz. Vortexer (Vortex-5) was obtained from Haimen Qilin Medical Instrument Factory (Jiangsu, China) and Refrigerated Centrifuge (CT-15RT) was obtained from Tianmei Biochemical Instrument Co. (Shanghai, China).

2.3. Synthesis of molecularly imprinted polymers

Phenylephrine (0.5 mmol) and MAA (4 mmol) were dissolved in methanol–acetonitrile (9 mL, 1:8, v/v). After self-assembly for 30 min, EDMA (20 mmol) and AIBN (0.36 mmol) were dissolved in the above solution for further reaction which was carried out in a thermostatic bath at 60 °C for 24 h. The obtained polymers were grinded and sieved to get particles in 38–54 μm, and washed with methanol–acetic acid (10:1, v/v) to remove both the template molecule and residual monomers. Then, the particles were rinsed sufficiently with deionised water and dried before use. As a control, non-imprinted polymers (NIPs) were synthesized simultaneously under the same procedure, but without the addition of the template molecule.

2.4. Sample pretreatment for ham sausages

The ham sausages purchased from the local markets in Baoding were spiked with SAL ranging from 1.2 to 600 ng g^{-1}. 3.0 g of each homogenized sample was put into a 10.0 mL conical tube and extracted by 4.0 mL of acetonitrile–ammonia (95:5, v/v) for 10 min under ultrasonic vibration. The extraction procedure was repeated once again with 3.0 mL acetonitrile–ammonia (95:5, v/v) and the supernatants obtained by centrifugation at 12,000 rpm for 5.0 min were combined together. The solution was concentrated to dryness at 50 °C, and then reconstituted with 1.0 mL of deionized water for further MISPE procedure.

2.5. MISPE conditions

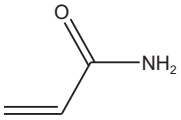
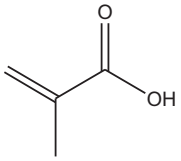
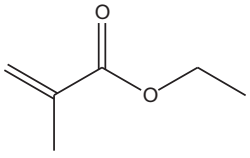
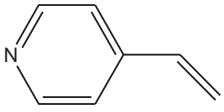
150 mg of MIP particles were packed into a 3.0 mL empty polypropylene cartridge with two glasswool frits at each end. The cartridge was consecutively preconditioned with 5.0 mL of methanol and 5.0 mL of water, followed by loading 1.0 mL of sample solution, then washed with 4.0 mL of methanol–water (3:7, v/v), and eluted with 3.0 mL of methanol–acetic acid (95:5, v/v). The eluent was evaporated to dryness and the residue was re-dissolved with 0.3 mL of mobile phase for further HPLC analysis.

3. Results and discussion

3.1. Synthesis of MIPs

In MIPs synthesis, the functional monomer and the solvent had evident influences on the imprinting effect. Six kinds of monomers were investigated in this study and the affinity of the resulted polymers to SAL were evaluated by a SPE procedure (Table 1), which indicated that the MIP using MAA as monomer showed higher recognition ability to SAL than the MIPs prepared using other monomers. This might be due to MAA has stronger electrostatic and hydrophobic interactions with target in polar environment. The carbonyl group of MAA may interact with phenylephrine by involvement of both its benzene hydroxy and hydroxy along the chain into hydrogen bonds, so as the hydroxyl group of MAA and secondary amine of phenylephrine. Although most MIPs were synthesized using analyte as template, it commonly suffered from

Table 1
Comparison of different phenylephrine-molecularly imprinted polymers.

Polymer no.	Monomer	Structure of monomer	Loss rate (%)
Polymer 1	AM		19.4
Polymer 2	MAA		0.4
Polymer 3	2-HEMA		21.6
Polymer 4	4-VP		14.5
Polymer 5	MAA + 4-VP	As above	6.7

template leaking in real sample application which affected the results of quantitative analysis. Therefore, in order to avoid the effect of template leakage and obtain the MIPs with special recognition ability to the SAL, dummy template was adopted to synthesize MIPs. Phenylephrine was chosen as the dummy template to prepare the MIPs due to its similar structures with SAL (benzene ring, hydroxy and amino-group). The quantity of methanol/acetonitrile in the polymerization mixtures had a critical effect on the pore properties and the surface area of the resulting polymers, because as a porogenic solvent, methanol/acetonitrile not only brought all the components into one phase but also created macropore structures in the imprinted polymers. The optimal composition of 9 mL methanol/acetonitrile (1:8, v/v) provided sufficient rigidity and desirable surface properties in the obtained MIPs.

Moreover, the ratios of template, monomer and cross-linker were investigated from 1:4:40 to 1:10:40 to get MIPs with satisfactory mechanical strength and affinity to SAL. The strength of the interactions presumably defined the subsequent affinity of the imprinted polymers, thus excessive MAA was needed to form more interaction sites with the template and increase the strength. The cross-linker was bifunctional monomers and one of their two double bonds reacted with the polymer chain while the other remained intact for further reaction. The reactions of the second double bond were crucial to form the cross-linking of the copolymer network which kept the imprinting cavities stable after the removal of template. As a consequence, the MIPs prepared at the molar ratio of 1:8:40 is suitable. The morphology of the MIPs evaluated by scanning electron microscope was shown in Fig. 2. It could be seen that the surface of polymers was rough and irregular. In the higher magnifications of 100,000, the rough porous and agglomerate surfaces were clearly observed that could create a large surface and space volume for SAL to embed into the cavity of the MIPs, resulting in its easier adsorption and elution.

3.2. Adsorption capacity of MIPs for SAL

Dynamic adsorption experiments for MIPs and NIPs were carried out to evaluate the adsorption capacity to SAL, which loading amount was ranged from 90 to 810 μg. Generally, the adsorption capacity of the MIPs increased with the increasing loading amount

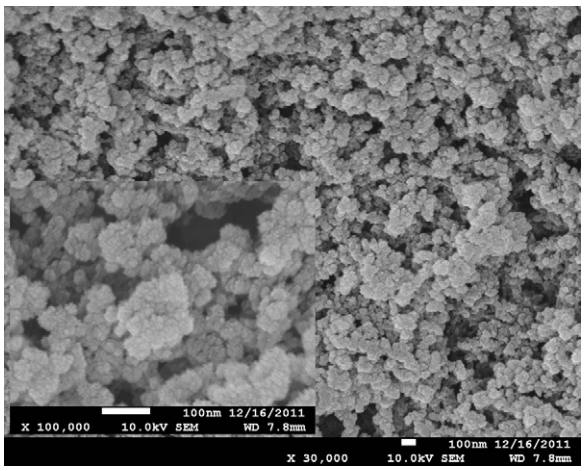


Fig. 2. Scanning electron microscopy of the MIPs.

in the initial stage, for the amount of analyte was not enough to saturate the specific binding cavities which formed by dummy template. When all the imprinted sites were occupied by the analyte, the adsorption capacity of the MIPs would reach the highest and keep saturated. However, the results of Fig. 3 showed that in the selected range of loading amount, the saturated adsorption capacity was not observed for MIPs which perhaps resulted from partially nonspecial adsorption. Moreover, dynamic adsorption showed that the MIPs had higher affinity and adsorption capacity to SAL than NIPs, which demonstrated the special imprinted recognition of the MIPs. However, the NIPs cannot form specific recognition sites to SAL due to the absence of the template (phenylephrine) in polymerization procedure. These results confirmed the presence of selective binding sites created by the template in the obtained MIPs and thus confirmed the successful imprinting processes via dummy template.

3.3. Optimization of the MISPE procedures

To evaluate the applicability of the MIPs for extraction and separation of trace level of SAL from complex samples, the general procedures of MISPE (precondition, loading, washing and elution) were optimized to achieve good selectivity and precision. Firstly, the cartridges were preconditioned with methanol and water, respectively. Then different loading solvents such as water, methanol, and acetonitrile–ammonia (95:5, v/v) were investigated,

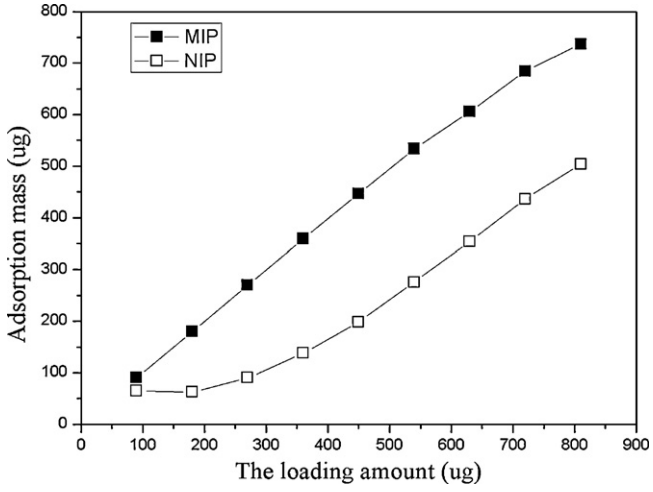


Fig. 3. Adsorption capacity of the MIP and NIP cartridges.

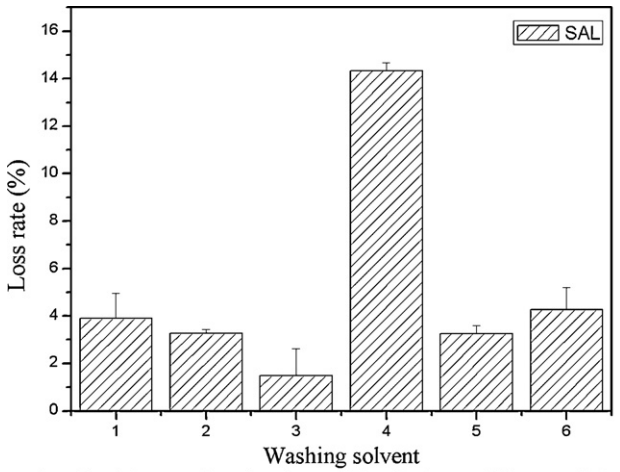


Fig. 4. Effect of washing solvents on the loss of SAL.

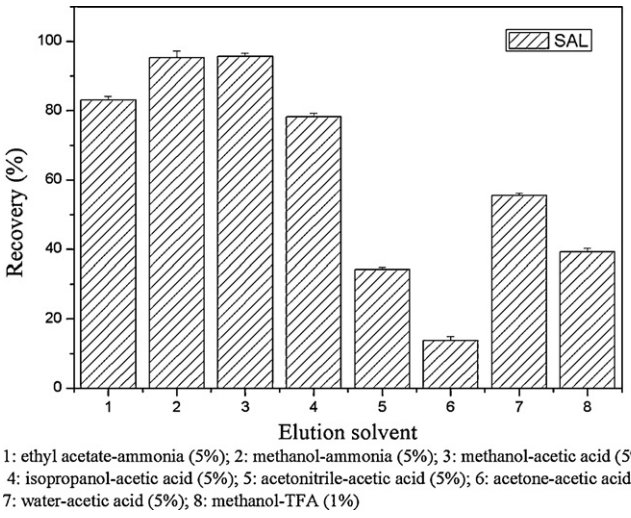


Fig. 5. Effect of elution solvents on recovery of SAL in MISPE.

and the results showed that almost all the loaded SAL was retained on MIPs and the loss rate was 0.3% when water was used as the loading solvent (50.5% and 36.4% when used acetonitrile–ammonia (95:5, v/v) and methanol, respectively). Thus, water was selected as the loading solvent.

The washing step was the most crucial procedure to maximize the special interactions between the analytes and binding sites, and to simultaneously decrease non-specific interactions with discard matrix components. Thus, different washing solvents such as methanol, acetone–chloroform (1:1, v/v), water, methanol–water (1:1, v/v), acetonitrile–water (1:1, v/v) and dichloromethane were investigated (Fig. 4). Although few analyte was lost from MIPs cartridge when water, acetone–chloroform (1:1, v/v) and dichloromethane used as the washing solvent, the purification efficiency of washing step was inapparent. In contrast, methanol–water (1:1, v/v) was sufficient to deliver the cleaner extract with the higher recovery of SAL. The effect of the proportions of methanol–water (1:1, 3:7, 4:6, v/v) was further discussed, and the lowest loss rate (1.0%) was obtained using methanol–water (3:7, v/v). For the purpose of minimum volume of washing solution to efficiently rinse the interferences, various volumes of methanol–water (3:7, v/v) ranged from 1.0 to 8.0 mL were investigated and the loss rate of SAL was almost constant (1.0–1.3%) with the volume from 1.0 to 6.0 mL, and then increased to 4.0% with the volume from 6.0 to 8.0 mL. Considering the loss rate, purification efficiency and economic factors, 4.0 mL of methanol–water (3:7, v/v) was chosen as the washing solution.

The strong imprint–analyte interaction must be destroyed to reach a high extraction recovery; therefore, different solvents termed of ethylacetate–ammonia (95:5, v/v), methanol–ammonia (95:5, v/v), methanol–acetic acid (95:5, v/v), isopropanol–acetic acid (95:5, v/v), acetonitrile–acetic acid (95:5, v/v), acetone–acetic acid (95:5, v/v), water–acetic acid (95:5, v/v) and methanol–TFA (99:1, v/v) were evaluated, and the results in Fig. 5 revealed that both methanol–ammonia (95:5, v/v) and methanol–acetic acid (95:5, v/v) provided the better recovery. Moreover, the cleaner chromatogram would be achieved when the latter was used as elution solvent. Therefore, different volumes of methanol–acetic acid in a range of 1.0–8.0 mL were investigated and the result showed that the recoveries obviously increased with the increasing volume from 1.0 to 3.0 mL, and then it almost constant even further increased the volume up to 8.0 mL. Considering the elution efficiency and solvent consumption, 3.0 mL was used as the optimum volume of elution solvent.

To compare the extraction efficiency of MIPs with other conventional sorbents, C_{18}, PCX, SCX, and HLB were also employed in the SPE procedures according to the previous reports [8,35–37]. Fig. 6 shows that the highest recovery (89.7%) was obtained by MIPs. Moreover, MIPs as SPE sorbent exhibited a cleaner chromatogram than other sorbents, which demonstrated the high selectivity and affinity of the MIPs to the target analyte. Additionally, it was worth noting that the MIP cartridge could be repeatedly used more than 10 times for clean-up of sausage samples with no noticeable deterioration in performance while other conventional sorbents lost their performance after one or two extractions.

3.4. Method validation

Calibration curve of SAL was constructed using the areas of the chromatographic peaks measured at nine increasing spiked levels, in a range of 1.2–600 ng g^{-1}. Each spiked level of samples was analyzed in triplicate. Good linearity was obtained for SAL in the concentration range and the calibration equation was $y = 492.36x + 9399$ with determination coefficient (r^2) of 0.9991. The limit of detection (LOD) and the limit of quantification (LOQ) calculated at the signal to noise ratio of 3 and 10 were 0.20 and 0.68 ng g^{-1}, respectively. The intra-day precision and accuracy of the method evaluated as RSD were ranged from 2.8 to 4.1% and the

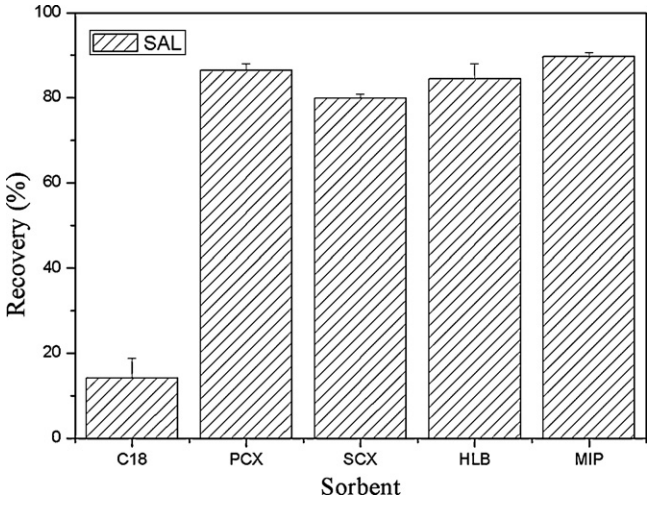


Fig. 6. Comparison of MIP with other sorbents.

Table 2
Recoveries of SAL in spiked ham sausages after MISPE.

Spiked levels	84 ng g^{-1}		330 ng g^{-1}		600 ng g^{-1}	
	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
SAL	86.5	2.7	96.9	2.2	85.7	3.6
	87.2	2.2	98.3	1.5	82.6	2.9
	85.9	1.6	100.5	3.0	87.2	2.3

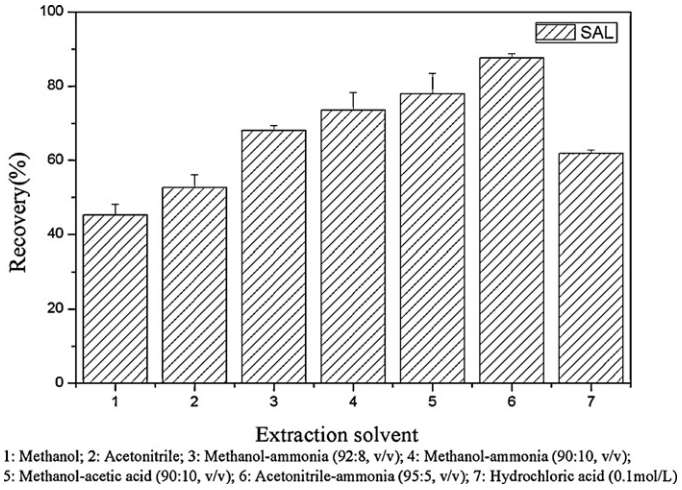


Fig. 7. Effect of extraction solvents on recovery of SAL.

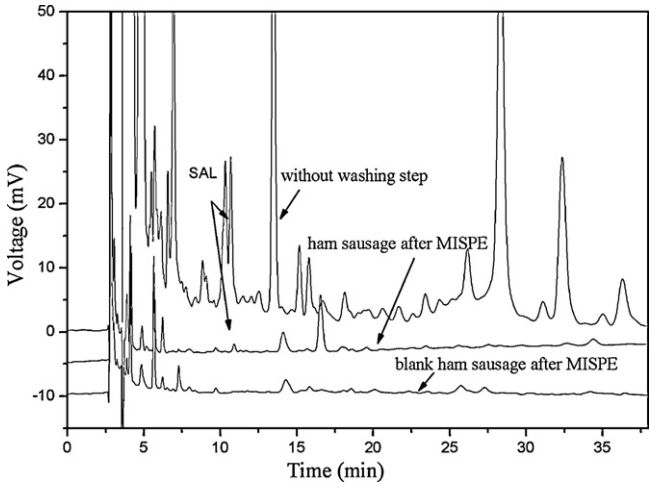


Fig. 9. Chromatograms of ham sausage samples after MISPE.

inter-day reproducibility was less than 6.3%. To study the effect of sample matrix and accuracy of the method, recovery experiments were carried out by spiking three levels of SAL in ham sausages (Table 2). The average recoveries for SAL at three spiked levels were in a range of 82.6–100.5% with RSD less than 3.6% (n = 3), which indicated that the method was reliable and could be used for the determination of trace SAL in complicated samples.

3.5. Analysis of real samples

Ham sausage products collected from the local markets were applied for validation of the MISPE–HPLC method. After homogenated by vortex, the ham sausage samples were pretreated using methanol, acetonitrile, methanol–ammonia (92:8, v/v), methanol–ammonia (90:10, v/v), methanol–acetic acid (90:10, v/v), acetonitrile–ammonia (95:5, v/v) and 0.1 mol/L hydrochloric acid, respectively. The highest recovery was obtained using acetonitrile–ammonia (95:5, v/v) as extraction solvent (Fig. 7). Therefore, 3.0 g of each ham sausage sample was pretreated using acetonitrile–ammonia (95:5, v/v) according to Section 2.4 and then followed by the MISPE–HPLC method. Three ham sausage samples were observed trace level of SAL in the range of 1.8 and 10.2 ng g^{-1}. The chromatograms (Figs. 8 and 9) of SAL revealed that the samples were significantly clean after being treated with the MISPE protocol and no genetic interferences from the ham sausage matrixes were observed.

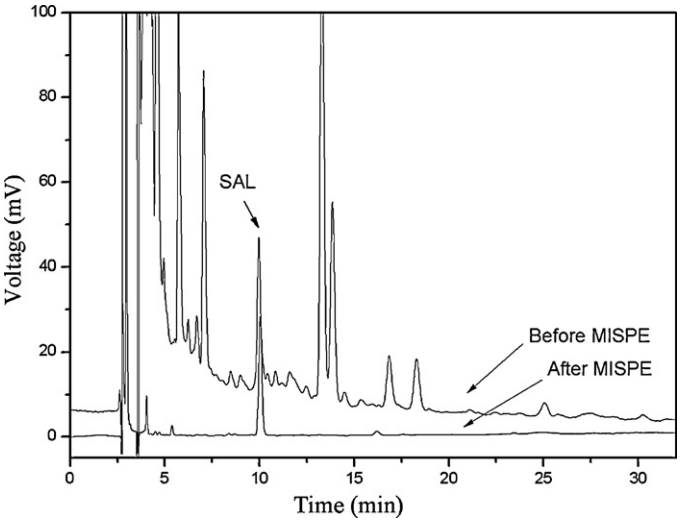


Fig. 8. Chromatograms of the spiked ham sausage before and after MISPE.

4. Conclusions

The new MIPs synthesized by bulk polymerization using phenylephrine as dummy template showed high affinity and adsorption capacity to SAL and it was successfully applied as a special SPE sorbent to overcome the drawbacks of template leakage of MIPs in quantitative analysis of ham sausages. The developed MISPE–HPLC method provides excellent selectivity and purification effect, and could be potentially applied for the determination of SAL in complicated food samples.

Acknowledgments

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References

[1] C.H. Qu, X.L. Li, L. Zhang, C.X. Xi, G.M. Wang, N.B. Li, H.Q. Luo, Chromatographia 73 (2011) 243.
[2] C.Y. Chai, G.Y. Liu, F. Li, X.F. Liu, B. Yao, L. Wang, Anal. Chim. Acta 675 (2010) 185.
[3] P. Zuo, Y. Zhang, J. Liu, B.C. Ye, Talanta 82 (2010) 1.

[4] J.D. Huang, Q. Lin, X.M. Zhang, X.R. He, X.R. Xing, W.J. Lian, M.M. Zuo, Q.Q. Zhang, Food Res. Int. 44 (2011) 92.
[5] M.Z. Zhang, M.Z. Wang, Z.L. Chen, J.H. Fang, M.M. Fang, J. Liu, X.P. Yu, Anal. Bioanal. Chem. 395 (2009) 2591.
[6] C. Widstrand, F. Larsson, M. Fiori, C. Civitareale, S. Mirante, G. Brambilla, J. Chromatogr. B 804 (2004) 85.
[7] J.R. Loss II., R.F. Orzechowski, R.S. Hock, Biomed. Chromatogr. 14 (2000) 1.
[8] S.H.R.A. Mazhara, H. Chrystyn, J. Pharm. Biomed. Anal. 50 (2009) 175.
[9] Q. Chen, L.Y. Fan, W. Zhang, C.X. Cao, Talanta 76 (2008) 282.
[10] S. Sirichai, P. Khanatharana, Talanta 76 (2008) 1194.
[11] W.Y. Wang, Y.L. Zhang, J.Y. Wang, X. Shi, J.N. Ye, Meat Sci. 85 (2010) 302.
[12] L. Wang, Y.Q. Li, Y.K. Zhou, Y. Yang, Chromatographia 71 (2010) 737.
[13] K.C. Yee, E. Lowe, G.A. Jacobson, Chromatographia 74 (2011) 355.
[14] M.W.F. Nielen, J.J.P. Lasaroms, M.L. Essers, J.E. Oosterink, T. Meijer, M.B. Sanders, T. Zuidema, A.A.M. Stolker, Anal. Bioanal. Chem. 391 (2008) 199.
[15] S.Y. Sheu, Y.C. Lei, Y.T. Tai, T.H. Chang, T.F. Kuo, Anal. Chim. Acta 654 (2009) 148.
[16] B.C. Somashekar, K. Basavaiah, J. Anal. Chem. 62 (2007) 432.
[17] B. Schuur, B.J.V. Verkuijl, J. Bokhove, A.J. Minnaard, J.G. Vries, H.J. Heeres, B.L. Feringa, Tetrahedron 67 (2011) 462.
[18] Q. Liu, J.B. Shi, L.X. Zeng, T. Wang, Y.Q. Cai, G.B. Jiang, J. Chromatogr. A 1218 (2011) 197.
[19] J. Du, H. Yan, D. She, B. Liu, G. Yang, Talanta 82 (2010) 698.
[20] H. Yan, B. Liu, J. Du, G. Yang, K.H. Row, J. Chromatogr. A 1217 (2010) 5152.
[21] M. García-López, P. Canosa, I. Rodríguez, Anal. Bioanal. Chem. 391 (2008) 963.
[22] T.F.S. Santos, A. Aquino, H.S. Dórea, S. Navickiene, Anal. Bioanal. Chem. 390 (2008) 1425.
[23] X.M. Cai, C.R. Wang, J.Y. Xu, X.Y. Xue, X.L. Zhang, X.M. Liang, J. Chromatogr. B 879 (2011) 657.
[24] J. Rubert, C. Soler, J. Manes, Talanta 85 (2011) 206.
[25] X. Wang, Y.Q. Wang, J.Q. Yuan, Q.L. Sun, J.H. Liu, C.C. Zheng, J. Chromatogr. A 1055 (2004) 135.
[26] S.M. Pourmortazavi, S.S. Hajimirsadeghi, J. Chromatogr. A 1163 (2007) 2.
[27] R.B. Ren, Y. Wang, R. Zhang, S.Q. Gao, H.Q. Zhang, A.M. Yu, Talanta 83 (2011) 1392.
[28] C.H. Ma, T.T. Liu, L. Yang, Y.G. Zu, S.Y. Wang, R.R. Zhang, Anal. Chim. Acta 689 (2011) 110.
[29] Z.H. Zhang, X. Yang, H.B. Zhang, M.L. Zhang, L.J. Luo, Y.F. Hua, S.Z. Yao, J. Chromatogr. B 879 (2011) 1617.
[30] P. Lucci, D. Derrien, F. Alix, C. Pérollier, S. Bayoudh, Anal. Chim. Acta 672 (2010) 15.
[31] S. Scorrano, L. Longo, G. Vasapollo, Anal. Chim. Acta 659 (2010) 167.
[32] Z.X. Xu, G.Z. Fang, S. Wang, Food Chem. 119 (2010) 845.
[33] M. Lasakova, P. Jandera, J. Sep. Sci. 32 (2009) 799.
[34] E. Turiel, A. Martín-Esteban, Anal. Chim. Acta 668 (2010) 87.
[35] J. Shan, C. Pan, J. Zhang, W. Niu, Acta Chromatogr. 20 (2008) 43.
[36] F. Caloni, M. Montana, C. Pasqualucci, G. Brambilla, G. Pompa, Vet. Res. Commun. 19 (1995) 285.
[37] K. Croes, P.T. McCarthy, R.J. Flanagan, J. Chromatogr. A 693 (1995) 289.