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Research article

Rapid extraction of domoic acid by a magnetic molecularly imprinted silica before HPLC measurement

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ABSTRACT

A magnetic molecularly imprinted silica solid was obtained by sol-gel polymerization for the separation of domoic acid. The solid showed rapid adsorption kinetics with an adsorption equilibrium time of 5 min. The solid showed affinity to domoic acid under the interference of tryptophan and could be repeatedly used for 5 times at least. The solid was used as a solid-phase-extraction sorbent for the extraction of domoic acid from clam samples before measurement with liquid chromatography. The detection limit of 0.20 mg kg^{-1} was lower than the allowable limits in several countries or areas. The recoveries in the spiked samples were 88% approximately.

1. Introduction

Domoic acid (DA) is a neurotoxin that causes amnesic shellfish poisoning. It is produced by algae and bioaccumulates in marine organisms such as shellfish, anchovies, and sardines. Exposure to this neurotoxin may cause short-term memory loss, brain damage, and, in severe cases, death in humans [1]. DA is highly toxic without an antidote available. New research has found that DA is heat-resistant and very stable, and can damage kidneys at concentrations 100 times lower than what causes neurological effects [2].

Detection methods based on HPLC were widely adopted for analyzing DA in shellfish [3, 4, 5, 6, 7]. However, complicated matrix in samples causes a long and tedious pretreatment process. Solid phase extraction (SPE) is a pretreatment method with merits of high recovery, little dosage, simple operation. But the selectivity of the traditional SPE is relatively poor. Molecularly imprinted polymers (MIP) have a good specificity for the target molecule. MIP is also highly stable and easy to prepare. So MIP has shown a vast application prospect in the field of separation-purification and sensors. Sellergren [8] was the first to develop a SPE method with MIP as absorbent with strong anti-interference ability.

Research work has been focused on the development of DA analytical method based on MIP. Lotierzo [9] et.al. first prepared an MIP sensor with photo-grafting onto a gold chip to achieve DA detection using surface Plasmon resonance. But high cost and toxicity restrict the popularization of this method. Nemoto et al. [10] used 1,3, 5-pentanetricarboxylic acid (PTA) as the template to prepare an MIP for the HPLC analysis of DA, which greatly lowered preparation cost and toxicity. Since then, PTA rather than DA was adopted to be the template for the preparation of MIP. For example, Zhou et al. [11] developed a quartz crystal microbalance MIP sensor to analyze DA in mussel. Dan et al. [12] fabricated an MIP-based phosphorescence sensor to analyze DA in shellfish. We [13] prepared an MIP through emulsion polymerization for the purification of clam samples to extract DA before its HPLC measurement. Zhou et al. [11] combined MIP and solid phase extraction (SPE) to develop an isolation method of DA from seafood samples.

Magnetic molecularly imprinted polymer (MMIP) can facilitate the isolation of trapped species from sample solution with a magnet. MMIP has stimulated growing interest in the pre-concentration or separation of analytes prior to detection [14, 15, 16]. But the articles about DA-MMIP are few [17]. We have prepared an MMIP by radical polymerization and used it for purification of DA in shellfish before HPLC detection [17]. The MMIP have a lower detection limit than MIP and C18 absorbent. But the equilibrium adsorption time was as long as 20 min. In this paper we presented an MMIP prepared by sol-gel method with high adsorption capacity and fast kinetics. The MMIP was applied as a SPE absorbent to the analysis of DA in clam with a satisfactory result.

2. Experiment details

2.1. Reagents and chemicals

All the chemicals were in analytical grade except for acetonitrile and methanol that were in chromatographic grade. DA was purchased from Sigma-Aldrich Reagent Co., Ltd. (Canada). PTA was purchased from

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Tokyo Chemical Industry (Tokyo, Japan). Tetraethyl orthosilicate (TEOS), 3-Aminopropyl triethoxysilane(APTES), trifluoroacetic acid and tryptophan (TRP) were obtained from Aladdin Reagent (Shanghai) Co., Ltd. (Shanghai, China). Polyethylene glycol 6000 (PEG), ethylene glycol (EG), polyvinyl pyrrolidone (PVP) were purchased from Xilong Chemical Co., Ltd. (Shantou, China). Acetonitrile, methanol and phosphoric acid were purchased from Tedia Company Incorporation (Fairfield, USA). High-purity water was prepared using a Millipore Simplicity Ultrapure water device (>18.0 MΩ cm, Millipore, Bedford, USA). Citric acid, phosphoric acid, triethylamine, ethylene glycol, polyethylene glycol, $FeCl_3{\cdot}6H_2O$, NaAc, HAc, and N,N-Dimethylformamide (DMF) were purchased from Xilong Chemical Co., Ltd. (Shantou, China). 4-vinylpyridine(4-VP), ethyleneglycol dimethacrylate(EDMA), and trifluoroacetic acid were obtained from Aladdin Reagent (Shanghai) Co., Ltd. (Shanghai, China).

2.2. Apparatus

A drying oven (DHG-9146A, Shanghai Jing Hong Laboratory Instrument Co., Ltd.) was used for hydrothermal preparation of magnetic Fe_3O_4. The morphologies of the MMIPs were measured using SEM (Hitachi Co., S4800, Japan). A Nicolet iS10 FT-IR spectrometer (Thermo Fisher Scientific Inc., USA) was used for infrared spectra scan with KBr pellets. A Lambda 265 UV-Vis spectrophotometer (Perkin Elmer Co, USA) was used to measure UV-Vis spectra. The dispersion treatment was operated with a KQ-500DA ultrasonic apparatus (Kunshan Instrument, Kunshan, China). The magnetic properties of the MMIPs were measured by VSM (Quantum Design, MPMS XL-7, America) at room temperature. Liquid chromatography separation was performed on an HPLC instrument (Agilent 1260 HPLC, USA).

2.3. Preparation of magnetic Fe_3O_4

Fe_3O_4 nanoparticles were prepared following a solvothermal method [18]. $NaAc{\cdot}3H_2O$ (7.2 g), and $FeCl_3{\cdot}6H_2O$ (2.7 g) were dissolved in EG (100 mL) under ultrasonic treatment. The solution (20 mL) was mixed with PEG (0.2 g). The mixture was sealed in a Teflon-lined stainless steel autoclave and was kept at 200 °C for 8h. After cooling to room temperature, the resulting black Fe_3O_4 precipitate was separated with a magnet, washed for 3 times with water (each 30 mL), and dried under vacuum. A suspension of Fe_3O_4 dispersed in water (20 mg mL^{-1}) was prepared for further use.

2.4. Silica modification of Fe_3O_4

Fe_3O_4 suspension (10 mL), PVP (1 g), water (20 mL) and ethanol (80 mL) were added to a 150-mL flask in sequence and the mixture was treated under ultrasound for 20 min. An ammonia solution (1.5 mL) was added to the mixture. The mixture was stirred (600 rpm) for 10min followed by the addition of a solution of TEOS (2 mL) and ethanol (40 mL). The mixture was kept stirring (600 rpm) for 24 h for silica polymerization. The black precipitate was separated with a magnet, washed with water for 3 times (each 30 mL), and dried under vacuum at 40 °C to yield $Fe_3O_4@SiO_2$.

2.5. Preparation of magnetic molecularly imprinted and non-imprinted silica

PTA (125 mg, 0.61mmol) and APTES (0.5 mL, 2.1 mmol) were dissolved in ethanol/water (V:V = 3 + 1, 15 mL). The solution was stirred for 20 min followed by the addition of TEOS (1 mL, 4.5mmol). The mixture was stirred for another 20 min. Then $Fe_3O_4@SiO_2$ (0.1 g) prepared in the previous step and HAc (1 mol/L, 0.1 mL) was added. The mixture was stirred (600 rpm) for 10 h for polymerization. The resulting black solid was separated magnetically. The elution of PTA template was carried out in a Soxhlet apparatus with methanol/acetic acid (V:V, 9 + 1) for 12h. The MMIP solid was washed with methanol for 12 h, dried under vacuum at 40 °C and sieved (200 mesh). Magnetic non-imprinted polymer (MNIP) solid was prepared following the same process but in the absence of PTA template.

2.6. Adsorption experiments

A DA standard solution (5mL) in acetonitrile/water (V:V, 19 + 1) was added to MMIP (or MNIP) absorbent (5mg). The mixture was shaken at room temperature for a certain time and was separated with a magnet. The solution was further centrifuged at 12500 rpm for 15 min and the DA concentration in the supernatant was measured by UV-vis at 242 nm. The adsorption capacity (Q) was calculated according to Eq. (1) and the imprinting factor (α) was calculated by Eq. (2).

$$Q=(C_0–C_e) \times V/m \quad (1)$$

$$\alpha = Q_{MMIP}/Q_{MNIP} \quad (2)$$

C_0 and C_e represent the initial and equilibrium DA concentration (μg·L^{-1}), respectively; V is the volume of the solution (mL); m is the weight of the absorbent (g). Q_{MMIP} and Q_{MNIP} represent the adsorption capacity (μg·g^{-1}) of MMIP and MNIP, respectively.

2.7. Reusability

One cycle of adsorption-elution experiment was carried out as follows: 1.5 mg of MMIP was mixed with 1.5 mL of DA solution in acetonitrile/water (V:V,19 + 1) at the concentration of 5.5 μg mL^{-1} in a centrifuge tube. After shaking bath for 10 min, the MMIP was magnetically collected and the solution was discarded. 1.5 mL of methanol-acetic acid (V:V,9 + 1) was added to the tube. After shaking bath for 10 min, the MMIP was magnetically collected and the eluate was discarded. The MMIP was treated with 1.5 mL of adsorption solvent, acetonitrile/water (V:V, 19 + 1), twice (each 5 min) to remove the remaining acetic acid.

2.8. Selectivity

The selectivity experiments were carried out at the presence of both DA and TRP in the solution. 2 mg of MMIP or MNIP was mixed with 1 mL of DA solution and 1 mL of TRP solution in acetonitrile/water (V:V,19 + 1) both at the concentration of 2.75 μg mL^{-1} in a centrifuge tube. After shaking bath for 10 min, the MMIP was magnetically collected and eluted with 1.5 mL of desorption solvent. The desorption process was repeated and the eluates were combined. The concentrations of DA and TRP in the eluates were analyzed with HPLC and the recoveries were calculated.

2.9. HPLC condition

Liquid chromatography separation was performed on an HPLC instrument (Agilent 1260 HPLC, USA) equipped with a diode array detector and an Eclipse Plus C18 column (4.6 × 100 mm, 3.5 μm). The mobile phase was a solution of water/acetonitrile (V:V, 85 + 15) with 0.1% trifluoroacetic acid. The flow rate and the injection volume were 0.5 mL min^{-1} and 100 μL, respectively. The wavelength of detector was 242 nm for DA and 280nm for TRP.

2.10. Extraction, purification and analysis of domoic acid in clam samples

Clam samples were obtained from a local supermarket. 5 g of the edible tissues were homogenized for 3 min at room temperature with a electric homogenizer (FS-1, made by Fuhua instrument Co. in China). 0.5 g of the of homogenate was mixed with 1 mL of methanol/water (V:V,1 + 1) and was extracted under ultrasound for 5 min. The mixture was centrifuged at 4000 rpm for 20 min, and the supernatant was transferred to a centrifuge tube. The slurry was ultrasonicated twice with 0.5 mL of methanol/water (V:V, 1 + 1) for 10 min. After centrifugation the

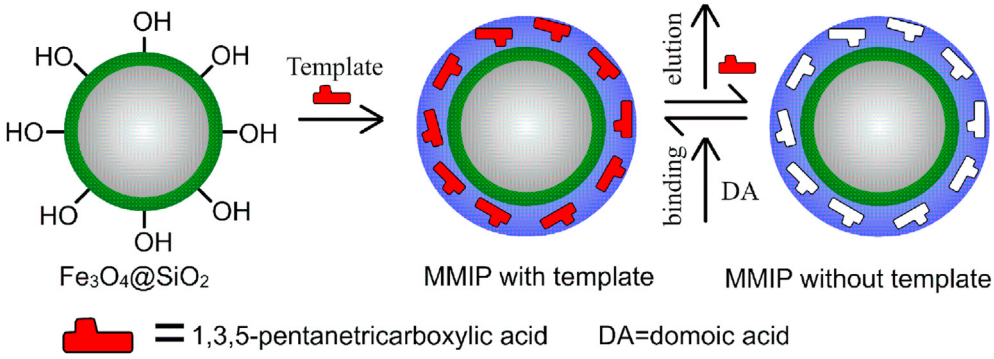

Figure 1. Scheme of reaction and purification process of MMIP.

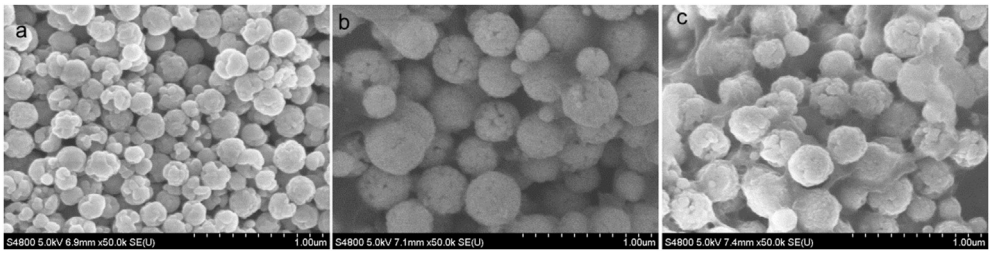

Figure 2. SEM images of Fe_3O_4 (a), MMIP (b), and MNIP (c).

obtained supernatant was also transferred to the centrifuge tube. Finally the supernatant was purged to nearly dryness with nitrogen and diluted to 1 mL with the adsorption solvent.

1 mL of the above extract was spiked with 1 mL of DA solution and 1mL of TRP solution at the same concentration to explore the selectivity of MMIP under the interference of TRP. 5 mg of MMIP (or MNIP) was mixed with 3 mL of the unspiked extract and the two spiked extract, respectively. The mixture was kept shaking for 10 min. The absorbents were magnetically separated, washed with 0.75 mL of acetonitrile/water (V:V, 1 + 1) and eluted with 0.75 mL of methanol/acetic acid (V:V, 1 + 9) twice. The replicate eluate were combined and evaporated to near dryness using nitrogen purging. The residues were dissolved in 1 mL of acetonitrile/water (V:V, 1 + 19) for HPLC analysis. The final concentrations of DA (or TRP) in the extract was 2.75 and 5.50 mg L^{-1}.

3. Results and discussion

3.1. Preparation

The reaction and purification process of MMIP was demonstrated in Figure 1. PTA was used as the template which was encapsulated in the imprinted silica layer. After elution, the template was removed to yield imprinted sites which exhibit selective to DA. The selective recognition was due to the spatial distance of the COOH groups on the PTA template molecules and the flexible conformation of the DA molecule as evidenced in reference. [10].

The effect of solvent was explored in the preparation of MMIP. The use of ethanol or acetonitrile leads to the MMIP product with low yield and polymer agglomeration. The adoption of water would increase the yield and polymer dispersity significantly. However excessive water

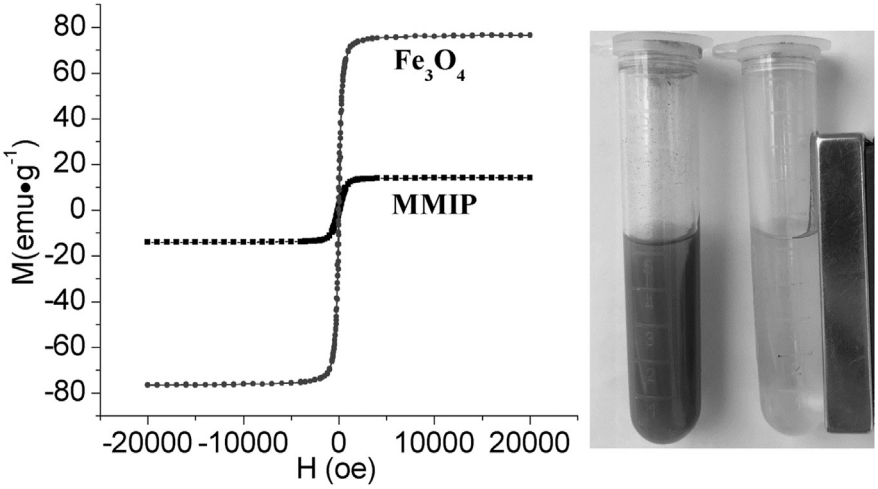

Figure 3. Magnetic induction curves of Fe_3O_4 and MMIP (left); Isolation of MMIP with an external magnet (right).

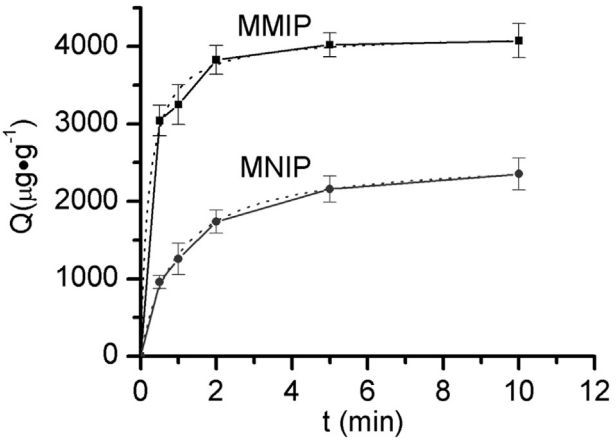

Figure 4. Adsorption kinetics curves of MMIP and MNIP. The dot lines represented the calculated curves according to the second-order kinetics equation.

would not favor the formation of binding sites and further decrease the selectivity of MMIP. It was found that the MMIP with the solvent of ethanol/water (V:V,3 + 1) exhibited a high adsorption amount (ca. 4000 μg g^{-1}) and imprinting factor (ca. 2.0), which were almost equivalent to those of MMIP with pure ethanol. Thus the solvent of ethanol/water (V:V,3 + 1) was chosen for the preparation of MMIP and MNIP.

3.2. Characterization

The SEM images are shown in Figure 2. Fe_3O_4, MMIP and MNIP particles were microspheres with regular shape and size. The particle sizes of MMIP and MNIP were a bit larger than Fe_3O_4, suggesting that the sol-gel imprinted layer was very thin, which make the mass-transfer of template molecules easier in the polymers.

Vibration sample magnetometer was applied to explore the magnetism of MMIP and Fe_3O_4. The results are shown in Figure 3 (left). It was found that MMIP and Fe_3O_4 particles had no obvious hysteresis and low magnetic coercivity, which was the characteristic of superparamagnet. The magnetic saturation induction of MMIP was only 14.0 emu·g^{-1}, much lower than the magnetic saturation induction of Fe_3O_4 (77.0 emu·g^{-1}). However, MMIP exhibited adequate magnetic response that the MMIP particles dispersed in solvent would be attracted rapidly together with an external magnet, as shown in Figure 3 (right). This magnetic response ability enabled the rapid and efficient separation of MMIP after extraction.

3.3. Adsorption

In the preparation of MMIP and MNIP, ethanol/water (V:V,3 + 1) was adopted as the solvent. It was found that adoption of the same solvent in adsorption would lead to slight difference in adsorption amount between MMIP and MNIP. The adoption of acetonitrile as adsorption solvent would enhance the selectivity, but DA was hardly dissolved in acetonitrile. After some trials we found that DA was soluble in a mixed solvent, acetonitrile/water (V:V,19 + 1). Furthermore, the solvent exhibited high selectivity as adsorption solvent.

The adsorption amounts of MMIP and MNIP with adsorption time at an initial DA concentration of 5.50 mg L^{-1} are shown in Figure 4. The MMIP exhibited adsorption kinetics similar to MNIP with an equilibrium time of only 5 min, which was much shorter than the value of 20 min in a DA-MMIP by radical polymerization [17]. The short equilibrium time implied that the imprinting shell was thin to enable the rapid and complete transfer of DA molecules from imprinted sites to solvent. The fact that the MMIP showed higher adsorption capacities than MNIP indicated that the binding sites on the surface of MMIP were more than that of MNIP.

Table 1. Desorption recoveries (%) of different solvents.

Times	MeCN–H_2O	H_2O	citric acid	MeOH-HAc
1st	None	8.5	61.7	68.6
2nd	None	10.9	67.9	74.5

Note: To improve the accuracy, the second eluate was combined with the first one before measurement.

The kinetics data of MMIP and MNIP were fitted using the non-linear fit tool in ORIGIN software. The fit result showed that the adsorption behavior could be evaluated using the second-order kinetics equation as $(Q_m - Q_t)^{-1} - Q_m^{-1} = k \cdot t$.

Q_m and Q_t represents the maximum adsorption amount and the adsorption amount at adsorption time of t, k represents the rate constant. The fit result provided the kinetics parameters of k and Q_m value as $3.8(0.6)\times10^{-6}$ mmol kg^{-1} min^{-1} and 13.36(0.28) mmol·kg^{-1} with a R^2 value of 0.9945 for MMIP, and $1.3(0.1)\times10^{-6}$ mmol kg^{-1} min^{-1} and 8.24(0.17) mmol·kg^{-1} with a R^2 value of 0.9971. The second-order kinetics equation implied that the adsorption rate was in relation to both DA concentration and binding site concentration.

3.4. Washing and desorption

Four solvents were chosen to explore their desorption ability for DA. The MMIP after absorption at 5.50 mg L^{-1} of DA solution was mixed with 0.75 mL of desorption solvents including acetonitrile-water (V:V,1 + 1), water, citric acid (0.5 mol L^{-1}) and methanol-acetic acid (V:V,9 + 1), respectively. Citric acid was the desorption solvent in a Chinese standard [19] and methanol-acetic acid (V:V,9 + 1) was a traditional solvent commonly used for the template removal in MIP. The mixtures were shaken for 5 min. The eluate was collected after the separation from MMIP absorbent with an external magnet, and was centrifuged for 15 min at 12500 rpm. The above desorption operation was repeated twice. The supernatants were measured with the UV-VIS method at 242nm. The desorption result is listed in Table 1. Acetonitrile-water (V:V,1 + 1) showed no elution ability while methanol-acetic acid (V:V,9 + 1) showed the highest desorption recovery. So acetonitrile-water (V:V,1 + 1) and methanol-acetic acid (V:V,9 + 1) were adopted as the washing and elution reagents, respectively.

3.5. Reusability

As can be seen from Figure 5, after 5 cycles of adsorption and elution, the MMIP exhibited high adsorption capacity for DA with only a 1.8% loss of adsorption capacity. This fact confirmed that the polymer network

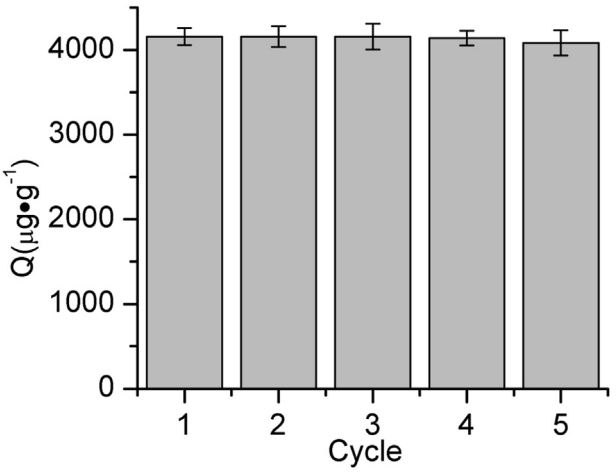

Figure 5. Adsorption amount of MMIP for DA at different times.

Table 2. The recoveries in the eluates of DA/TRP mixture ($n = 3$).

Sample	DA Recovery (%)	TRP Recovery (%)
MMIP	75.8 ± 2.8	41.3 ± 3.6
MNIP	50.1 ± 3.3	36.6 ± 5.1

Table 3. Recoveries of MMIP and MNIP in clam samples.

Spiked concentration ($mg{\cdot}L^{-1}$)	Recovery (%) of MMIP	Recovery (%) of MNIP
2.75	87.6 ± 7.0	55.4 ± 7.6
5.50	88.3 ± 6.2	57.1 ± 6.9

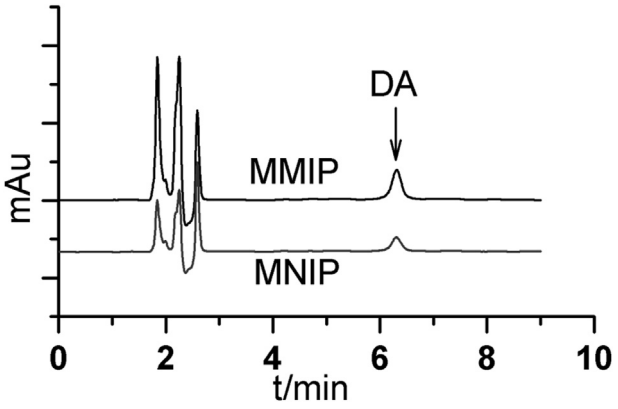

Figure 6. HPLC diagrams of the spiked extracts (2.75 mg L^{-1}) under the treatment of MMIP and MNIP, respectively.

and the imprinting sites in MMIP basically remain intact after repetitious elution and adsorption.

3.6. Selectivity

As a component of shellfish tissue, TRP would interfere the analysis of DA in HPLC method. The selectivity experiments were carried out at the presence of both DA and TRP in the same solution after treated with 2 mg of sorbents. The result was listed in Table 2. After elution, the DA and TRP recoveries of MMIP were both higher than those of MNIP, which confirmed that the MMIP exhibited selectivity for DA and TRP due to the presence of binding sites. In addition, DA could be fully absorbed by MMIP and eluted under the interference of TRP. So MMIP had higher affinity to DA than to TRP.

3.7. Sample analysis

The HPLC diagrams and the analysis results were listed in Figure 6 and Table 3 respectively. Most of the DA in the sample was extracted after treatment with MMIP, and the recoveries of MMIP were higher than MNIP. However, the recoveries of MMIP were about 90%, which was probably caused by the interference of impurities in the extract. The linear range was from 0.1 to 6.2 mg L^{-1}, and the detection limit ($3\sigma/K$, $n = 9$) for DA was calculated to be 0.20 mg kg^{-1} this was much lower than the permitted level of 20 mg kg^{-1} in shellfish tissue issued by Canada, European Union and the USA [20]. The MMIP was anticipated to be a promising material for the extraction of DA in real samples due to the low detection limit and rapid adsorption behaviour.

3.8. Comparison

Magnetic sorbents currently reported for DA extraction were listed in Table 4 to show their properties. It can be seen that the MMIP in this work exhibited high adsorption, rapid adsorption and anti-interference ability.

4. Conclusion

In this work, an MMIP material was synthesized through the sol-gel method. The MMIP exhibited high affinity and rapid adsorption for DA. The maximum adsorption amount and the adsorption equilibrium time were measured to be 13.36(0.28) $mmol{\cdot}kg^{-1}$ and 5 min. The MMIP was used as a SPE sorbent to extract DA selectively. The adsorption, washing and elution reagents were determined. Acetonitrile/water (V:V,19 + 1), acetonitrile-water (V:V,1 + 1) and methanol-acetic acid (V:V,9 + 1) were adopted as the adsorption, washing and elution reagents, respectively. The MMIP featured good repeated usage and anti-interference ability in comparison with other works and was applied in the clam sample pretreatment before HPLC determination. The detection limit was as low as 0.20 mg kg^{-1}. The analytical results indicated that the MMIP was a suitable material for the extraction of DA in aquatic products with spiked recoveries about 88%.

Declarations

Author contribution statement

Zhiyong Huang: Conceived and designed the experiments.
Shengyang Chen, Lei Li: Performed the experiments.
Aihong Peng: Analyzed and interpreted the data.
Zhengzhong Lin: Conceived and designed the experiments; Wrote the paper.

Table 4. Comparison of current magnetic sorbents for DA extraction.

ref	Sorbent	Q_{max}[d] $\mu g{\cdot}g^{-1}$	t_{eq}[e] (concentration)	Interference test	Analysis method
[17]	Fe_3O_4@MIP[a]	1600	30min(1 mg L^{-1})	Trp	HPLC–DAD[f]
[21]	$CuFe_2O_4$ nanospheres	-	<1min (50 μg L^{-1})	-	HPLC–MS
[22]	Fe_3O_4@SiO_2@UiO-66[b]	4300	4h (50 μg L^{-1})	-	HPLC–MS
This work	Fe_3O_4@MIP[c]	4155	5min(5.5 mg L^{-1})	Trp	HPLC–DAD

[a] MIP was constructed via organic monomer and crosslinker.
[b] UiO-66 was a metal organic coordination polymer with tetrahedral and octahedral cavities.
[c] MIP was constructed via inorganic sillica framework.
[d] Q_{max} represented the maximum adsorption amount that was obtained from adsorption isotherms.
[e] t_{eq} represented the adsorption equilibrium time that was obtained from adsorption kinetics. The concentration was the original DA concentration in adsorption kinetics.
[f] DAD represented the diode array detector.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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