Talanta 85 (2011) 2100–2105

Contents lists available at ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Determination of dicofol in aquatic products using molecularly imprinted solid-phase extraction coupled with GC-ECD detection

Hui Wang[a], Hongyuan Yan[a,b,*], Mande Qiu[b], Jindong Qiao[a], Gengliang Yang[a]

[a] Key Laboratory of Pharmaceutical Quality Control of Hebei Province & College of Pharmacy, Hebei University, Baoding 071002, China
[b] Key Laboratory of Medicinal Chemistry and Molecular Diagnosis, Hebei University, Baoding 071002, China

ARTICLE INFO

Article history:
Received 15 April 2011
Received in revised form 7 July 2011
Accepted 10 July 2011
Available online 23 July 2011

Keywords:
Molecularly imprinted microspheres
Dummy template polymerization
Solid-phase extraction
Dicofol
Aquatic products

ABSTRACT

The novel molecularly imprinted microsphere (MIM) that could be applied as special sorbent was synthesized by aqueous suspension polymerization using 1,1-bis(4-chlorophenyl)-1,2,2,2-tetrachloroethane (α-chloro-DDT) as the dummy template. The obtained MIM exhibited good recognition and selectivity to dicofol and it was successfully applied as selective sorbent of solid-phase extraction for the determination of dicofol from aquatic products. At the optimum conditions of the molecularly imprinted solid-phase extraction (MISPE) coupled with GC-ECD, good linearity for dicofol was achieved in a range of 0.4–100 ng g^{-1} (r^2 = 0.9995) and the recoveries at three spiked levels were ranged from 85.8% to 101.2% for aquatic products with the relative standard deviation (RSD) less than 5.6%. The presented MISPE-GC-ECD method exhibited the advantages of simplicity, selectivity and sensitivity, and could be potentially applied to the determination of dicofol in complicated aquatic products.

1. Introduction

Dicofol [2,2,2-trichloro-1,1-bis(4-chlorophenyl)ethanol] is a non-systemic organochlorine acaricide that is used on a worldwide variety of fruit, vegetables, ornamentals, teas, and field crops, simultaneously as the substitute for the forbidden organochlorine pesticides such as dichlorodiphenyltrichloroethane (DDT) [1,2]. However, with its structural similarity and biological activity to DDT, the toxicity, capacity for endocrine disturbance, and carcinogenicity of dicofol also have a strong influence on the environment and human health by biological accumulation effect [3,4]. Currently, more and more countries are concerned about its continuing use, and the residues of dicofol in grain, vegetables, fruit and animal-derived food are limited strictly. Recently, the emergency recall in Japan towards the Chinese-made eel due to the high amount of dicofol residue further revealed the serious pollution status. Therefore, it is important to focus on the development of a simple, accurate and sensitive analytical method for determination of dicofol in aquatic products.

At present, the common methods for the determination of dicofol are mainly gas chromatography-electron capture detector (GC-ECD) [5,6], gas chromatography–mass spectrometry (GC–MS) [7,8] and high performance liquid chromatography (HPLC) [9]. Owing to the complexity of sample matrices and low levels of analytes, sample pretreatment and enrichment process become the crucial steps in the analytical procedure. Until now, several pretreatment process, such as liquid–liquid extraction (LLE) [10], solid-phase extraction (SPE) [11], matrix solid-phase dispersion (MSPD) [12], supercritical fluid extraction (SFE) [13], single-drop microextraction (SDME) [14], microwave-assisted steam distillation (MASD) [15] and enzyme-linked immunosorbent assay (ELISA) [16] have been suggested. Among them, SPE is one of the most important and frequently used pretreatment methods for either matrix simplification or trace enrichment because of its matured technology and the good adaptability towards multiple matrix samples [17]. However, conventional SPE sorbents (C_{18}, C_8, silica gel, etc.) usually based on non-specific hydrophobic interactions that lead to the co-extraction of interfering compounds, thus preventing the reliable quantification of analytes from complex samples. Therefore, development of new sorbents with high affinity, specific recognition and high stability is of great significance.

Molecular imprinting technology is a synthetic approach to produce functionalized materials having specific molecular recognition properties for a given compound, its analogues, or for a single enantiomer [18–21]. Molecularly imprinted polymers (MIPs) show many outstanding advantages, such as predetermined recognition ability, chemical and thermal stability, relative ease and low cost of preparation. Therefore, they have become increasingly attractive as class- or compound-specific sorbents and recently, a number of publications have shown the effectiveness of the MIPs as highly selective SPE sorbents for the isolation and

* Corresponding author. Tel.: +86 312 5971107; fax: +86 312 5971107.
E-mail address: yanhy@hbu.edu.cn (H. Yan).

0039-9140/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.talanta.2011.07.061

preconcentration of traces analytes such as triazines [22], methimazole [23], zearalenone [24], dichlorvos [25], betulin and betulinic acid [26], water-soluble acid dyes [27] in environmental or biological samples. The application of these synthetic polymers as sorbents allows the analytes of interest to be pre-concentrated while simultaneously removing interfering compounds from the matrix, so that selective enrichment and cleanup are obtained, resulting in a higher accuracy and a lower detection limit in the subsequent analysis [28–31]. However, the work done so far on the aspect of dicofol residue in aquatic products is quite scarce. In addition, most MIPs are synthesized by using the target analyte as template showing high selectivity but serious templates leaking, which would affect the results of quantitative analysis in real sample application. Therefore, several research on the adoption of dummy template (always be one of the structural analogues of target analyte) for the synthesis of MIPs have been reported [32–34], since Andersson et al. [35] described the dummy imprinting procedure for the first time to eliminate the effect of template leaking on quantitative analysis.

In this work, new molecularly imprinted microsphere (MIM) was synthesized using α-chloro-DDT as dummy template and applied as SPE sorbent for the selective separation and quantitative determination of dicofol residue in fish and prawn products. Various factors affecting the preconcentration and separation of the analyte were discussed in detail and the applicability of this method was also evaluated.

2. Experimental

2.1. Chemicals

Dicofol and 1,1-bis(4-chlorophenyl)-1,2,2,2-tetrachloroethane (α-chloro-DDT) were obtained from Dacheng Pesticide Co., Ltd. (Shandong, China). Polyvinylpyrrolidone (PVP), chloroform, dichloromethane, ethyl acetate and hexane were obtained from Huaxin Chemical Co. (Baoding, China). Methacrylic acid (MAA), methanol, acetonitrile, ethanol, acetone, acetic acid, and 2,2-azobisisobutyronitrile (AIBN) were purchased from Kermel Chemical Co. Ltd. (Tianjin, China). Ethylene glycoldimethacrylate (EGDMA) was purchased from Sigma–Aldrich (St. Louis, MO, USA). All the other reagents used in the experiment were of the highest grade commercially available. Double deionized water was filtered with 0.45 μm filter membrane before use.

2.2. Instrumentation and conditions

The chromatographic analysis was carried out on a Shimadzu GC-2014 system equipped with a split/splitless injector and an ECD-2014 detector (Shimadzu, Japan). High purity nitrogen (99.999%) was used as carrier gas. The capillary column was DB-5 (5% phenyl-methylpolysiloxane as stationary phase, 30 m × 0.53 mm × 1 μm) from Agilent Co. (Wilmington, DE, USA) and its flow rate was set at 6 mL min^{-1} with a split ratio of 1.0. An N-2000 chromatographic workstation (Zheda Zhineng, Hangzhou, China) was used as the data acquisition system. The temperature programmed mode was as follows: the oven temperature was 220 °C and held for 12 min. The injection port and detector temperatures were set at 280 °C and 310 °C, respectively. The injection volume was 1 μL for all the solutions. A vortex oscillator (Vortex-5, Qilin Medical Instrument, Jiangsu, China) was used to assist the liquid extraction of the aquatic product.

2.3. Synthesis of the imprinted microspheres

The imprinted microspheres were prepared by aqueous suspension polymerization. Firstly, 3 g of polyvinylpyrrolidone was dissolved into 120 mL of water. The solution was poured into a 250 mL flanged reactor flask in a water bath (60 °C) and then was stirred at 600 rpm under a nitrogen stream. Secondly, precomplexation was obtained by mixing α-chloro-DDT (0.157 g), MAA (0.69 mL), EGDMA (9.4 mL) and AIBN (0.250 g) with 20 mL chloroform and sonicating for 5 min to maintain homogeneity. Then the solution was added dropwise to the above PVP solution. Finally, the flask with all the reagents still maintained in the water bath with stir for 24 h for polymerization. The produced suspension was filtered and the MIM was washed with methanol–acetic acid (9:1, v/v) to remove the template and unreacted monomers. A non-imprinted microsphere (NIM) was synthesized by the similar procedure in the absence of template molecules.

2.4. Sample preparation of aquatic products

All the aquatic products were obtained from the local supermarket in Baoding. 5 g of the minced samples, determined to be free of the analyte, were spiked with 0.2 μg of dicofol in conical flask. After 30 min rest in the dark, 25 mL *n*-hexane which was used to extract the analyte was added. The mixture was consumingly vortexed for 5 min at room temperature and soaked for 30 min, then 1 g sodium chloride was added and vortexed for another 1 min to precipitate the protein. Subsequently, the extract was centrifuged at 4000 rpm for 5 min and the clarification solution was collected for further SPE procedure.

2.5. Procedure of MISPE

The empty polypropylene cartridge (5 cm × 8 mm I.D.) was preplaced a polyethylene frit (0.2 μm) at its bottom and then packed into 300 mg of the MIM. After attaching with another frit on the top end, the MISPE cartridges were pretreated with 5 mL chloroform, methanol and hexane, respectively, followed by loading 25 mL of sample extract. Then the MISPE cartridges were washed by 4 mL of hexane–acetone (19:1, v/v) and eluted by 4 mL of methanol–acetic acid (9:1, v/v). The eluents were evaporated to dryness under vacuum at 25 °C, and the residues were reconstituted into 0.3 mL of hexane for further GC-ECD analysis.

3. Results and discussion

3.1. Preparation of the imprinted microspheres

At present, the most previous MIPs are synthesized by using the target analyte as template and show high selectivity. However, due to the fact that the residual template molecules embedded in the polymer are hard to be rinsed away completely, these MIPs always exhibit serious templates leakage, which would affect the results of quantitative analysis seriously in real sample application. To avoid the effect and obtain the imprinted materials with special recognition ability to dicofol, adoption of dummy template is considered. Dicofol is structurally similar to DDT. It differs from DDT by the replacement of the H on C-1 by OH functional group. But considering that the DDT is forbidden with high dangers towards organisms, we find another suitable one which includes structures of both compounds to be disguised as the template for the synthesis of MIMs providing high affinity towards dicofol. Therefore, α-chloro-DDT (one of the structural analogues of dicofol) is adopted as dummy template to synthesize the MIM by aqueous suspension polymerization using MAA, EGDMA and PVP as monomer, cross-linker and dispersant, respectively. Referring to the previous reports on the application of MIM [36–38], several factors affecting the morphology of the molecularly imprinted microspheres are optimized.

Generally, a large excess of functional monomer versus the template would improve the stability of the pre-polymerization

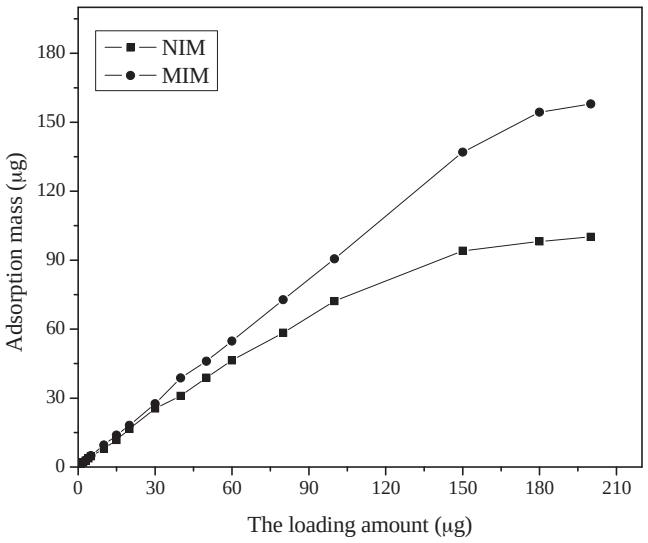
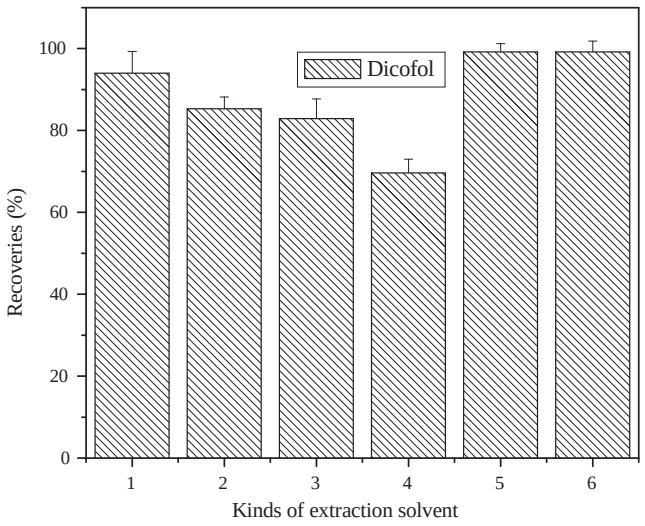


Fig. 1. Adsorption capacity of the MIM and NIM cartridges towards dicofol.



(1: Acetone; 2: Ethanol; 3: Acetonitrile; 4: Ethylacetate; 5: Hexane-saturated acetonitrile; 6: Hexane)

Fig. 2. Effect of extraction solvents on the recovery of dicofol.

complex by shifting the association–dissociation equilibrium towards complex formation. And the amount of cross-linker should be high enough not only to maintain the mechanical stability and rigidity of the polymer matrix, but also to ensure a certain flexibility to make the functional monomer and template enter the cavities easily, thus resulting in a higher binding capacity. In this work, the molar ratios of template/monomer/cross-linker were investigated and 1:4:25 was selected to ensure the formation of defined recognition sites with polymer. In addition, the type and volume of porogenic solvent have obvious effect on both the solubility of template–monomer mixture and the morphology of polymers. Herein, 20 mL of chloroform was chosen as the porogenic solvent because it ensured good solubility of the template and favoured hydrogen bonds between template and monomer molecules. Moreover, to further improve the recognition of the obtained MIM in aqueous samples, the MIM was prepared using PVP as dispersant in 120 mL of water, which exhibited good mechanical strength and special affinity to analytes.

3.2. Characteristics of MIM

The morphology of the MIM evaluated by scanning electron microscope indicated the MIM were monodispersed and uniformly sized with diameter distribution from 30 μm to 60 μm. Moreover, the majority of the MIM were spherical, and the surface of the MIM was porous and rough, which was suitable for rebinding or releasing the target molecules from its surface. The binding capacity of MIM and NIM evaluated by dynamic adsorption was shown in Fig. 1. The adsorption data showed MIM offered a higher affinity to dicofol than NIM, which demonstrated the binding affinity of the MIM was mainly from the specific sites formed by the imprinting effect.

Apart from these recognition properties of MIM, the materials also exhibited high physical and chemical resistance towards various external degrading factors, such as mechanical stress, elevated temperatures and extreme chemical medium. Thus, they could be stored for a long time and reused for many times without loss of "memory effect". The MISPE catridges could be regenerated in excess up to 100 times [24,39,40], being superior to the commercial single-used SPE which could be used once only. Additionally, in comparison with immunoaffinity materials which were based on the similar principle of specific, reversible interaction between antibody and antigen, and might be recycled 0–60 times [41–44], the MIM exhibited longer lifetime, better stability and ease synthesis methods. In this work, the MISPE catridges were reused throughout the entire experiment without any loss in efficiency.

3.3. Extraction process

The selection of extraction solvent was critical in obtaining a satisfactory recovery of the target analyte from the complex sample matrix. In this study, 5 g of minced samples spiked with dicofol were extracted by ethyl acetate, acetone, *n*-hexane, ethanol, acetonitrile and acetonitrile-saturated hexane, respectively, as described above. The best recovery of analyte was found among hexane-saturated acetonitrile and *n*-hexane extract (Fig. 2). However, chromatograms of the extract obtained by hexane-saturated acetonitrile showed more interferences than *n*-hexane. Further experiment compared these two extracts as loading solution directly towards the MISPE cartridges and the results revealed that 82.1% of dicofol was washed away by hexane-saturated acetonitrile in the loading step. This might be that acetonitrile with high polarity had a direct disruption for the specific interaction between the template molecule and the MIM, which led to a significant loss in loading step for MISPE cartridge. Considering all the factors above, *n*-hexane was selected as extraction solvent and further loading solvent in this study.

3.4. Optimization of the MISPE procedures

In order to evaluate the applicability of the MIM for separation and determination of dicofol by GC-ECD, general parameters for MISPE including the type and amount of washing solution and eluent were optimized. The washing solvent was one of the crucial factors to maximize the specific interactions between the analytes and binding sites, and to simultaneously decrease non-specific interactions to discard matrix components from the cartridge. Therefore, 25 mL spiked *n*-hexane extract was loaded into the cartridge and washed by 3 mL of acetonitrile, methanol, water–methanol (1:1, v/v), chloroform, hexane–acetone (9:1, v/v), water, water–acetonitrile (1:1, v/v), hexane and hexane–acetone (19:1, v/v), respectively. The chromatograms obtained from the eluents revealed that chloroform and acetonitrile were sufficient to discard the impurities from the sample matrix, but more than 90% of the target analyte were washed out simultaneously (histogram in Fig. 3). Water, water–acetonitrile (1:1, v/v) and hexane created

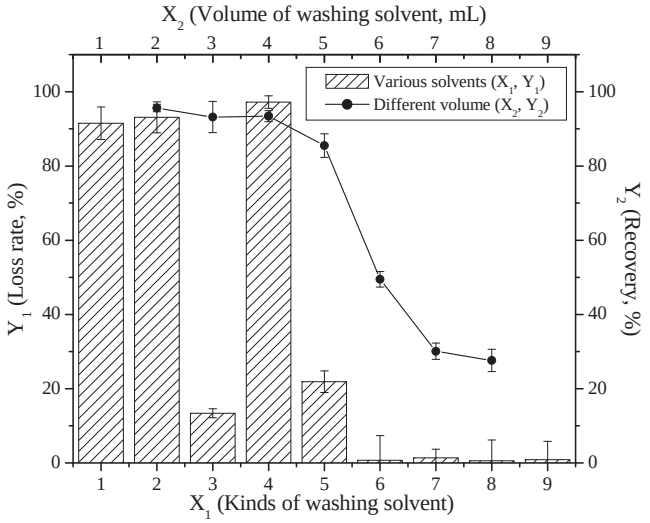


Fig. 3. Effect of washing solvents and volume on extraction efficiency of dicofol.

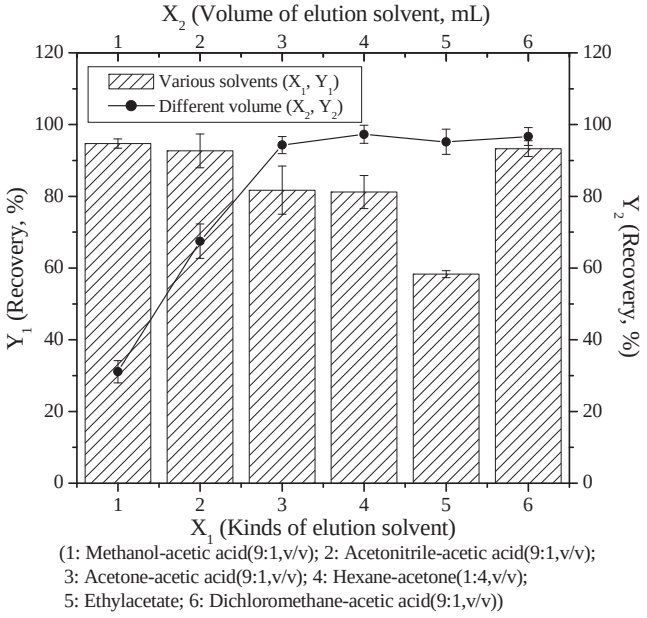


Fig. 4. Effect of elution solvents and volume on the recovery of dicofol.

little loss of analyte, but the purifying effect of the washing step was inapparent. Comprehensive considering the recoveries and purification efficiency, hexane–acetone (19:1, v/v) was selected as the washing solvent which exhibited the best ability of eluting the matrix interferences with low loss rate of dicofol (0.9%).

For the purpose of determining the minimum volume of washing solution able to efficiently rinse the interferences, recoveries of docofol were studied in applying different hexane–acetone (19:1, v/v) volumes of 2–8 mL (line graph in Fig. 3). The results showed that the recovery of dicofol reduced slightly from 95.6% to 93.4% with the increase volume of hexane–acetone (19:1, v/v) from 2 mL to 4 mL, and then reduced sharply to 30.1% with the further increase to 8 mL. Considering that less solvent could not remove the interferences sufficiently from the MISPE cartridges, while excess solvent could result in the loss of the analyte, thus, 4 mL of hexane–acetone (19:1, v/v) was chosen as the washing solution in further work.

An appropriate eluent should be selected to ensure the analyte can be eluted sufficiently from the MISPE cartridge. For this purpose, 3 mL of different type of solvents including methanol–acetic acid (9:1, v/v), acetonitrile–acetic acid (9:1, v/v), acetone–acetic acid (9:1, v/v), hexane–acetone (1:4, v/v), ethylacetate and dichloromethane–acetic acid (9:1, v/v) were applied. Methanol–acetic acid (9:1, v/v) as elution solvent offered the best efficiency (histogram in Fig. 4). Subsequently, by comparison of the obtained data of different volumes of elution solvent ranged from 1 mL to 6 mL, the best recovery of dicofol was achieved at 4 mL of methanol–acetic acid (9:1, v/v), and more volume just provided almost constant recovery values (line graph in Fig. 4). Based on the elution efficiency and solvent consumption, 4 mL of methanol–acetic acid (9:1, v/v) was adopted to ensure a quantitative elution of the analyte from the sorbent.

3.5. Analytical parameters

The method was validated for linearity, precision, repeatability, recovery, detection limits, inter-assay and intra-assay deviation. Calibration curve using MISPE-GC-ECD was constructed by least-squares linear regression analysis of the peak area versus analyte concentration using seven increasing dicofol concentrations, in the range of 0.4–100 ng g^{-1}. A good linearity equation $Y = 7.43 \times 10^4 X + 9.89 \times 10^4$ was established throughout the concentration range for the dicofol with the correlation coefficient (r^2) = 0.9995. The accuracy and precision were assessed by performing replicate analyses of spiked samples in five replicates in the same day and consecutive three days. The intra-day precision and accuracy of the method evaluated as RSD were ranged from 2.2% to 3.6% and the inter-day reproducibility was below 6.3%. Based on a signal-to-noise ratio of 3 and 10, the limit of determination (LOD) and the limit of quantitation (LOQ) of dicofol was 0.1 ng g^{-1} and 0.4 ng g^{-1}.

In regard to selectivity and extraction efficiency, the MIM had also been compared with NIM and other conventional sorbents such as silica [8], alumina [10] and C_{18} [13] in the SPE procedures according to the previous reports. Fig. 5 shows the recoveries of dicofol for the five sorbents under their respective optimized conditions, as well as the NH_2-SPE [11] and florisil [45]. Among them, MIM provided the highest recovery (95.3–98.7%). And under the same experimental condition, 54.6% of the analyte was flowed out in the washing step when using NIM as SPE sorbent, which

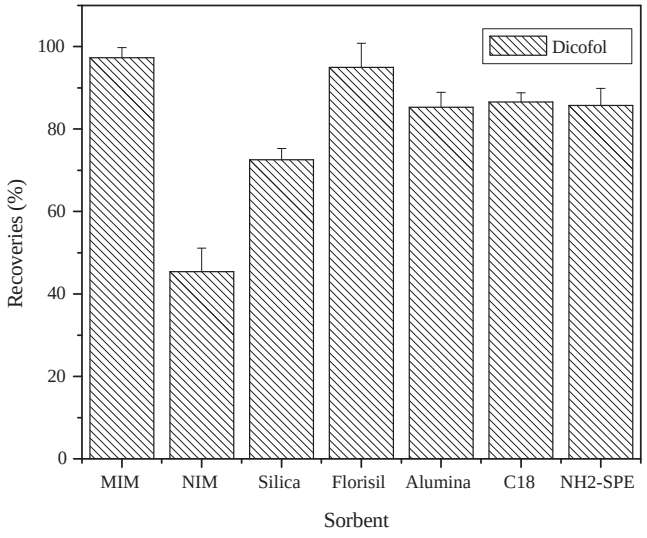


Fig. 5. Comparison of MIM with other sorbents.

Table 1
Comparison of merits of methods for determination of dicofol.

Matrix/mass of sample (g)	Sample preparation/ volume of solvent (mL)	LOD (ng g^{-1})	Recovery (%)[b]	RSD (%)	Detection	Ref.
Apple/100	LE, CC[a]/635	5	93–96.4	1.3	GC-ECD	[10]
Orange pulp/0.5	MSPD/20	70	90–95	2.6–9	GC-ECD	[12]
Orange peel/0.5		80	87–94	5–7.6		
Soil/6	SFE, C_{18}-SPE/16	<10	81–83	5.3–6.6	GC-ECD	[13]
Soil/25	LE/110	5	>80	<7		
Tea/2	MASD/30	0.2	110.1	4.7	GC-ECD	[15]
Fish/10	LE, NH_2SPE/49	1	78.9–86.9	7.4–9.7	GC-MS	[11]
Fish/1	LE, CC/66	3	70–120	<10	GC-ECD	[45]
Fish/5	LE, MISPE/33	0.1	85.8–101.2	5.6	GC-ECD	Present work

[a] CC: column chromatography.
[b] Recovery (%): recovery of spiked samples.

demonstrated the higher affinity of MIM towards dicofol, and further proved that the binding affinity of the imprinted polymer was mainly from the specific sites formed by the imprinting effect.

Moreover, the comparison of the MISPE-GC-ECD method with other methods for determination of dicofol is shown in Table 1. Under the detection system of GC-ECD, high sensitivity was achieved by using relatively low sample consumption and organic solvent. Simultaneously, in comparison to the national standard of China [45] in which traditional column chromatography containing florisil as sorbents was adopted with large consumption of organic or dangerous inorganic solvents (hexane, dichloromethane, concentrated sulfuric acid) for tedious sample preparation processes, the proposed method simplified the whole procedure significantly with higher selectivity and sensitivity.

3.6. MISPE of real sample application

To demonstrate the potential of MISPE for the selective clean up of the analyte in real samples, six aquatic products (grass carps, wuchang fish, carp, crucian carp, eel, prawn) collected from the located markets were analyzed. As a result, dicofol was detected from wuchang fish as 5.2 ng g^{-1} with RSD about 2.6% which was below the maximum residual limit (MRL) established by Japan (10 ng g^{-1}). The chromatogram obtained of wuchang fish using MISPE-GC-ECD method is shown in Fig. 6.

Moreover, the recovery study was then carried out by spiking the grass carps with three different levels of dicofol under the optimized condition. The results obtained are shown in Table 2. The

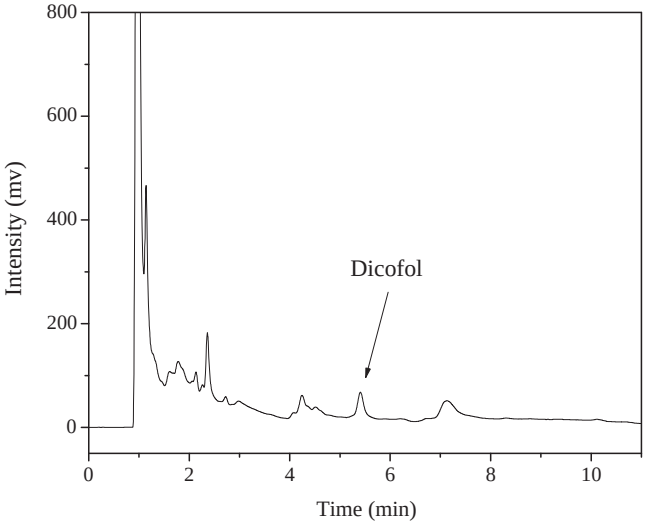


Fig. 6. Chromatogram of the wuchang fish samples.

Table 2
Recoveries of the MISPE-GC-ECD method for spiked grass carps (n = 3).

Analytes	Added (ng g^{-1})	Founded (ng g^{-1})	Recovery (%)	RSD (%)
Dicofol	1	0.99	99.2	2.9
	5	5.06	101.2	4.6
	10	8.58	85.8	5.6

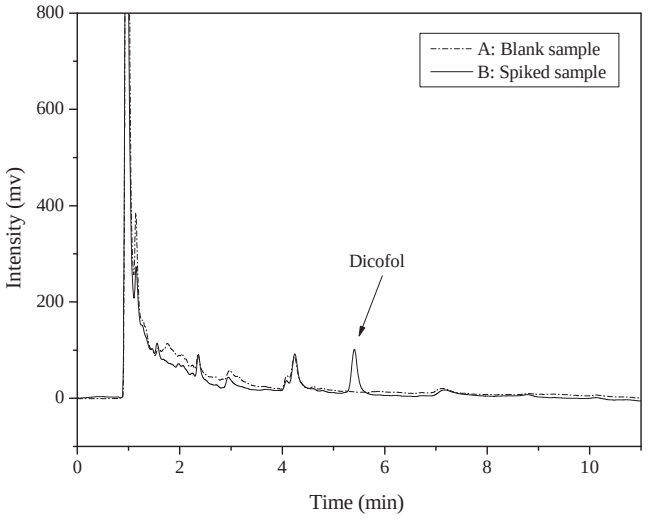


Fig. 7. Chromatograms of the grass carp samples. (A: blank sample, B: spiked sample: 10 ng g^{-1}, injection volume: 1 μL.)

average recoveries for the analyte were in the range of 85.8–101.2% with RSD less than 5.6%. The potential interferences of fish matrix were also investigated by extracting and analyzing five blank fish samples by MIM. The chromatograms (Fig. 7) of the elution fractions revealed that the samples were significantly cleaner after the MISPE protocol and demonstrated the absence of endogenous interfering compounds from the fish matrixes at the retention time of the analyte. The results above further proved the reliability and accuracy of the developed MISPE-GC-ECD method for the determination of dicofol in aquatic products.

4. Conclusions

In this work, the novel MIM synthesized by aqueous suspension polymerization using α-chloro-DDT as dummy template showed good recognition and affinity to dicofol and was successfully applied as SPE sorbent to selectively extract it from the aquatic products. Under the optimum condition, coupled with the adoption of large volume for sample loading towards the MISPE cartridge, high sensitivity with the LOD of 0.1 ng g^{-1} was achieved by using relatively low sample consumption and organic solvent com-

pared with other methods. The presented MISPE-GC-ECD method exhibited the advantages of simplicity, rapidity, sensitivity and accuracy, and could be potentially applied for the monitoring of dicofol residue in aquatic products.

Acknowledgements

The project was sponsored by National Natural Science Foundation of China (20905019, 21011140338), and Natural Science Foundation of Hebei Province (B2010000209).

References

[1] X. Qiu, T. Zhu, B. Yao, J. Hu, S. Hu, Environ. Sci. Technol. 39 (2005) 4385–4390.
[2] Y. Fujii, K. Haraguchi, K.H. Harada, T. Hitomi, K. Inoue, Y. Itoh, T. Watanabe, K. Takenaka, S. Uehara, H.R. Yang, M.Y. Kim, C.S. Moon, H.S. Kimh, P.Y. Wang, A.P. Liu, N.N. Hung, A. Koizumi, Chemosphere 82 (2011) 25–31.
[3] P.F. Hoekstra, B.K. Burnison, A.W. Garrison, T. Neheli, D.C.G. Muir, Chemosphere 64 (2006) 174–177.
[4] J. Zhang, H. Yan, T. Yang, Y. Zong, J. Environ. Manage. 92 (2011) 53–58.
[5] S. Jaggi, C. Sood, V. Kumar, S.D. Ravindranath, A. Shanker, J. Agric. Food Chem. 49 (2001) 5479–5483.
[6] S.R. Rissato, M.S. Galhiane, F.R.N. Knoll, B.M. Apon, J. Chromatogr. A 1048 (2004) 153–159.
[7] A.G. Frenich, M.J. Gonzalez-Rodrıguez, F.J. Arrebola, J.L.M. Vidal, Anal. Chem. 77 (2005) 4640–4648.
[8] M. Saitta, G.D. Bella, F. Salvo, S.L. Curto, G. Dugo, J. Agric. Food Chem. 48 (2000) 797–801.
[9] S. Han, J. Qiao, Y. Zhang, L. Yang, H. Lian, X. Ge, H. Chen, Chemosphere 83 (2011) 131–136.
[10] S.B. Singh, I. Mukherjee, J. Maisnam, P. Kumar, M. Gopal, G. Kulshrestha, J. Agric. Food Chem. 57 (2009) 11277–11283.
[11] S. Chen, X. Yu, X. He, D. Xie, Y. Fan, J. Peng, Food Chem. 113 (2009) 1297–1300.
[12] M.L. Ribeiro, J.R. Amador, L. Polese, E.F.G. Jardim, E.V. Minelli, O.C.P.D. Cordis, J. Agric. Food Chem. 48 (2000) 2818–2820.
[13] S.R. Rissato, M.S. Galhiane, B.M. Apon, M.S.P. Arruda, J. Agric. Food Chem. 53 (2005) 62–69.
[14] M. Zhang, J. Huang, C. Wei, B. Yu, X. Yang, X. Chen, Talanta 74 (2008) 599–604.
[15] J. Ji, C. Deng, H. Zhang, Y. Wu, X. Zhang, Talanta 71 (2007) 1068–1074.
[16] S. Hongsibsong, T. Prapamontol, C. Suphavilai, J. Wipasa, M. Pattarawarapan, W. Kasinrerk, Hybridoma 29 (2010) 495–500.
[17] D.M. Pavlovic, S. Babic, D. Dolar, D. Asperger, K. Kosutic, A.J.M. Horvat, M. Kastelan-Macan, J. Sep. Sci. 33 (2010) 258–267.
[18] H. Yan, K.H. Row, G. Yang, Talanta 75 (2008) 227–232.
[19] H. Yan, F. Qiao, K.H. Row, Anal. Chem. 79 (2007) 8242–8248.
[20] A. Beltran, F. Borrull, P.A.G. Cormack, R.M. Marce, Trends Anal. Chem. 29 (2010) 1363–1375.
[21] F.G. Tamayo, E. Turiel, A. Martin-Esteban, J. Chromatogr. A 1152 (2007) 32–40.
[22] R. Koeber, C. Fleischer, F. Lanza, K.S. Boos, B. Sellergren, D. Barcelo, Anal. Chem. 73 (2001) 2437–2444.
[23] M. Pan, J. Wang, G. Fang, W. Tang, S. Wang, J. Chromatogr. B 878 (2010) 1531–1536.
[24] P. Lucci, D. Derrien, F. Alix, C. Perollier, S. Bayoudh, Anal. Chim. Acta 672 (2010) 15–19.
[25] Z. Xu, G. Fang, S. Wang, Food Chem. 119 (2010) 845–850.
[26] B. Claude, C. Viron-Lamy, K. Haupt, P. Morin, Phytochem. Anal. 21 (2010) 180–185.
[27] X. Luo, Y. Zhan, X. Tu, Y. Huang, S. Luo, L. Yan, J. Chromatogr. A 1218 (2011) 1115–1121.
[28] C. He, Y. Long, J. Pan, K. Li, F. Liu, J. Biochem. Biophys. Methods 70 (2007) 133–150.
[29] E. Caro, R.M. Marce, F. Borrull, P.A.G. Cormack, D.C. Sherrington, Trends Anal. Chem. 25 (2006) 143–154.
[30] M. Curcio, F. Puoci, G. Cirillo, F. Iemma, U.G. Spizzirri, N. Picci, J. Agric. Food Chem. 58 (2010) 11883–11887.
[31] J. Haginaka, J. Sep. Sci. 32 (2009) 1548–1565.
[32] X. Feás, J.A. Seijas, M.P. Vázquez-Tato, P. Regal, A. Cepeda, C. Fente, Anal. Chim. Acta 631 (2009) 237–244.
[33] J. Wang, R. Guo, J. Chen, Q. Zhang, X. Liang, Anal. Chim. Acta 540 (2005) 307–315.
[34] H. Zhang, T. Song, W. Zhang, W. Hua, C. Pan, Bioorg. Med. Chem. 15 (2007) 6089–6095.
[35] L.I. Andereeson, A. Paprica, T. Arvidsson, Cromatographia 46 (1997) 57–62.
[36] K. Yoshimatsu, K. Reimhult, A. Krozer, K. Mosbach, K. Sode, L. Ye, Anal. Chim. Acta 584 (2007) 112–121.
[37] L. Ye, K. Mosbach, React. Funct. Polym. 48 (2001) 149–157.
[38] L. Ye, R. Weiss, K. Mosbach, Macromolecules 33 (2000) 8239–8245.
[39] O. Ramström, R.J. Ansell, Chirality 10 (1998) 195–209.
[40] Y. Tan, H. Peng, C. Liang, S. Yao, Sens. Actuators B 73 (2001) 179–184.
[41] N. Delaunay, V. Pichon, H. Marie-Claire, J. Chromatogr. B 745 (2000) 15–37.
[42] A.E. Prince, T.S. Fan, B.A. Skoczenski, R.J. Bushway, Anal. Chim. Acta 444 (2001) 37–49.
[43] F. Kondo, Y. Ito, H. Oka, S. Yamada, K. Tsuji, M. Imokawa, Y. Niimi, K. Harada, Y. Ueno, Y. Miyazaki, Toxicon 40 (2002) 893–899.
[44] T.M. Phillips, J. Biochem. Biophys. Methods 49 (2001) 253–262.
[45] Ministry of Agriculture of China, Determination of dicofol residues in aquatic products by gas chromatography, SC/T 3040-2008 (2008) (China).