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ORIGINAL PAPER

Facile one-pot synthesis of magnetic molecular imprinting polymers as a novel adsorbent for the enrichment of imidacloprid based on a magnetic dispersive micro-solid-phase extraction in water samples

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Abstract

The pesticide residues have become one of the issues in the fields of environmental health and food safety. Herein, an effective method was successfully developed to rapidly determine imidacloprid in environmental water samples based on magnetic molecular imprinted polymers (MMIP) coupled with high-performance liquid chromatography. MMIP were creatively synthesized in one-pot synthesis step via the nucleation process of Fe_3O_4 magnetic nanoparticles and the polymerization process of molecular imprinted polymers simultaneously, using imidacloprid as template and dopamine as functional monomer. The parameters effected on the extraction efficiency, including pH value and the amount of the adsorbent, have been optimized. Under the optimal conditions, MMIP was used to extract imidacloprid in water samples with excellent recovery (88–97.3%) and precision (1.2–3.3%). Therefore, magnetic nanoparticles-based dispersion solid-phase extraction could be a good candidate for highly efficient extraction of imidacloprid without complicated operations.

Keywords Adsorption · Imidacloprid detection · Magnetic molecular imprinted polymers · One pot

Introduction

Imidacloprid is an efficient contact neonicotinoid insecticide. It causes a blockage of the nicotinergic neuronal pathway. Imidacloprid was the most widely used insecticide in the world to prevent, control, or eliminate pests on crops. Presently, the increase usage of imidacloprid is due to boosting the productivity highly in modern agriculture (Fujii et al. 2019; Tao et al. 2019). However, excessive and improper use of imidacloprid also could cause wide spread contamination in surrounding, even cause the food safety issues severely (Li et al. 2019). Owing to the high solubility of imidacloprid in natural water systems, imidacloprid may cause a huge danger to human health and environmental safety (Sultana et al. 2018; Si et al. 2018; Wang et al. 2015). In addition, imidacloprid may damage the DNA of human peripheral lymphocytes even at trace levels (Sultana et al. 2018). Therefore, the monitoring of pesticide residues is of great significance to satisfy the demand for human health and to protect the ecosystem.

Various analytical methods sprang up in the last decades for the determination of imidacloprid, including enzyme-linked immunosorbent assay (Yan et al. 2012), capillary electrophoresis (Sanchez-Hernandez et al. 2014), electrochemical sensor (Ben Brahim et al. 2016), high-performance liquid chromatography (HPLC) (Mandic et al. 2005), HPLC–mass spectrometry (HPLC–MS) (Haroune et al. 2015; Zhang et al. 2018), and gas chromatography (GC) (Chen et al. 2017). Instrument-based analysis methods mentioned above could not accurately determine the quantity of imidacloprid by poor purification or direct instrument detection, because the environment matrix is very complex (Liu et al. 2015). Hence, sample pretreatment technology in pesticide residue detection is particularly important.

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Solid-phase extraction was first discovered in the early 1970s and developed rapidly as a high-efficiency sample pretreatment technology (Kollmann and Brennan 1979).This method is developed to supplement or replace liquid–liquid extraction, which is widely used for residue analysis of pesticides, veterinary drugs and other harmful substances in environmental protection and food security field (Reinholds et al. 2019; Wang et al. 2019a, b). Compared with the traditional method, this method has the advantages of good reproducibility, time saving, and less solvent consumption. Recently, the technology of magnetic nanoparticles-based dispersion solid-phase extraction has been developed rapidly. Magnetic solid-phase extraction (MSPE) has received much attention due to its merits of environment friendliness, rapid separation process, excellent adsorption efficiency, and easily automated assay (Capriotti et al. 2019).

Molecular imprinted polymers (MIP) have high selectivity and affinity for a specific analyte or a group of structure-related compounds by forming molecular recognition holes matching template molecules in shape, size, and functional groups (Mirzajani et al. 2017; Wang et al. 2013). Magnetic molecular imprinted polymers (MMIP) have the advantages of both magnetic responsive and the high affinity of MIP. MMIP adsorbent for imidacloprid was prepared for selective separation of imidacloprid from honey and vegetable samples (Kumar et al. 2018). A novel and simple imprinting route based on graphene was proposed to fabricate an electrochemical sensor for sensitive and selective determination of imidacloprid residue (Zhang et al. 2017). Under the condition of the additional magnetic field, analytes can be separated directly and selectively. MMIP showed very superior performance in the field of trace pollutant's extraction and separation, and gradually became one of the most promising fields in sample pretreatment.

However, the preparation of MMIP normally contains several reaction steps, including the magnetic nanomaterial nucleation process and molecular imprinting polymerization process. The step-by-step preparation method of MMIP material undoubtedly brings the problems of the complexity of preparation, and of the uncontrollability of material performance. To this end, a novel MMIP was synthesized based on dopamine as functional monomer, and imidacloprid as template molecules in one reaction step in the presence of ferrous salt in an alkaline environment (Fig. 1). The obtained MMIP can adsorb imidacloprid with high efficiency and can be separated in the additional magnetic field easily.

Materials and methods

Reagents and apparatus

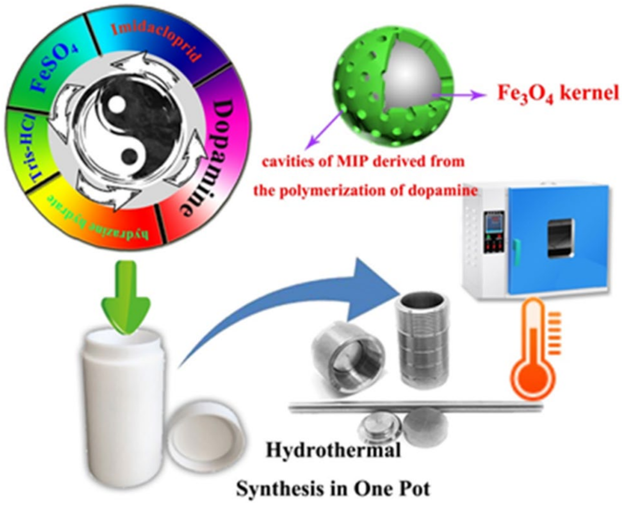

Fig. 1 One-pot synthesis of magnetic molecular imprinting polymers

All chemicals were obtained from a commercial seller and used of analytical grade or better without further purification. Ferrous sulfateheptahydrate ($FeSO_4 \cdot 7H_2O$), hydrazine hydrate ($N_2H_4 \cdot H_2O$), dopamine, methanol, ethanol, dichloromethane, and tri-hydroxymethyl amino methane hydrochloride (Tris–HCl) were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). Imidacloprid, triazophos, triazolone, methomyl, carbaryl, and chlorpyrifos were obtained from the department of agriculture environmental protection monitoring, China. The organic solvents used for chromatographic analysis were HPLC-grade water and methanol (Fisher Scientific Inc.).

Imidacloprid was diluted in pure methanol to obtain a stock solution at the concentration of 1000 μg/mL. The stock solution of imidaclopridwere stored in refrigerator at 4 °C. The working solutions were prepared daily by diluting with methanol: water (1:1). The stock was used to prepare spiking solutions for the method validation and for the recovery study, as well as for the calibration standards for analysis by HPLC–UV.

The morphology of the polymers structures was determined by a FEI Emission Scanning Electron Microscope (SEM, SU8200, Hitachi, Japan) with an acceleration voltage of 10 kV. Infrared absorption spectrum tests were conducted on a fourier-transform infrared spectroscopy (FTIR, Nicolet iS10 FTIR Spectrometer, Thermo Scientific, USA). HPLC analysis was performed on a Rigol L-3000 system. Separation of imidacloprid was achieved on a C-18 column (5 μm particle size, 250 × 4.6 mm) and the temperature was set at 40 ℃. A mixture of 70% solvent A (methanol of chromatographic purity) and 30% solvent B (ultrapure water) was used as the mobile phase. The flow rate of the mobile phase was set at 1 mL/min. The sample injection volume was 10 μL. The retention time of imidacloprid chromatography peak was at 2.84 min under the chromatography detection condition mentioned above.

Synthesis of magnetic molecular imprinted polymers

A novel MMIP was synthesized based on dopamine as functional monomer. The significant merit of the MMIP preparation methodology is processing the nucleation and polymerization procedures into one step into one pot. The synthesis processes of MMIP were declared briefly as follows.

0.2 mmol of $FeSO_4·7H_2O$ was dissolved in 3 mL of ultra-pure water. The solution was transferred into a teflon beaker. 0.2 mL hydrazine hydrate was added. Then, 0.1 mmol of imidacloprid and 0.25 mmol of Tris–HCl was dissolved in 25 mL of ultra-pure water. Adjusted the sollution to pH = 8.5 before transferred the mixture into the teflon beaker contained ferrous salt. Lastly, 0.3 mmol of dopamine was added into the reactor. Heated the reactor gradually to 150 ℃ and maintained heating state for 5 h.

The reactor was then cooled to ambient temperature in 2 h. The product was separated by a magnet, and the supernatant was decanted. The products were washed with ultra-pure water and methanol for several times. The MMIP was obtained after the freeze drying. The MMIP material was kept into the referigrator at − 20 ℃ before usage. Because the stability and reusability of magnetic material was excellent under the storage conditions of drying and low temperature.

Extraction procedure

50 mg MMIP adsorbent was incubated into the 25 mL of imidacloprid solution at the concentration of 800 μg/L (pH = 6.5). After that, the adsorbent in the suspension was separated by a magnet and dried slightly by nitrogen flow. Eluent of the imidacloprid from the MMIP was accomplished by washing the adsorbent with 500 μL of organic solvents by fierce vortex for 1 min in 3 times. Finally, 100 μL of the eluent was drawn out and directly injected into the HPLC for analysis. The procedure of the extraction of imidacloprid by MMIP is illustrated in Fig. 2.

Results and discussion

Characterization of MMIP

Magnetic non-molecular imprinted polymers (MNIP) and MMIP, were characterized by FTIR and SEM. The FTIR spectra of pure Fe_3O_4 (a), MNIP (b) and MMIP (c) shown the distinct differences each other (Fig. 3A). The band at 627 cm^{-1} of curve b and c, may be attributed to the Fe–O–Fe vibration of pure magnetite Fe_3O_4. The bands at 810 cm^{-1} were possibly related to C–C groups with skeletal vibration, and the bands at 1493 cm^{-1} were possibly related to C=C groups with skeletal vibration. The bands at 2463 cm^{-1} could indicate the unsaturated C bonds on the benzene ring stretching. Compared with the FTIR transmission bands of Fe_3O_4, MNIP and MMIP, it was obviously seen that dopamine was well-combined with the Fe_3O_4 microspheres forming the composites in MMIP.

The morphology of the obtained Fe_3O_4, MNIP and MMIP composites were identified by SEM (Fig. 3B–D). The SEM image of pure Fe_3O_4 particles in Fig. 3B. The SEM image of MNIP in Fig. 3C shown a high crystallinity of Fe_3O_4, which is in a large crystal size at about 20 μm with the structure of trigonal prism. There was more poly-dopamine enriched on the surface of MMIP than that of MNIP (Fig. 3D). Meanwhile, there are cavities in the poly-dopamine surface so that MMIP can adsorb imidacloprid specifically.

Adsorption efficiency of MMIP

To evaluate the adsorption efficiency of the different magnetic adsorbents toward the target analysts, the adsorption ability of Fe_3O_4, MNIP and MMIP to imidacloprid were compared, respectively (Fig. 4a). The MMIP exhibited a higher affinity to imidacloprid because of the more imidacloprid adsorbed onto the MMIP.

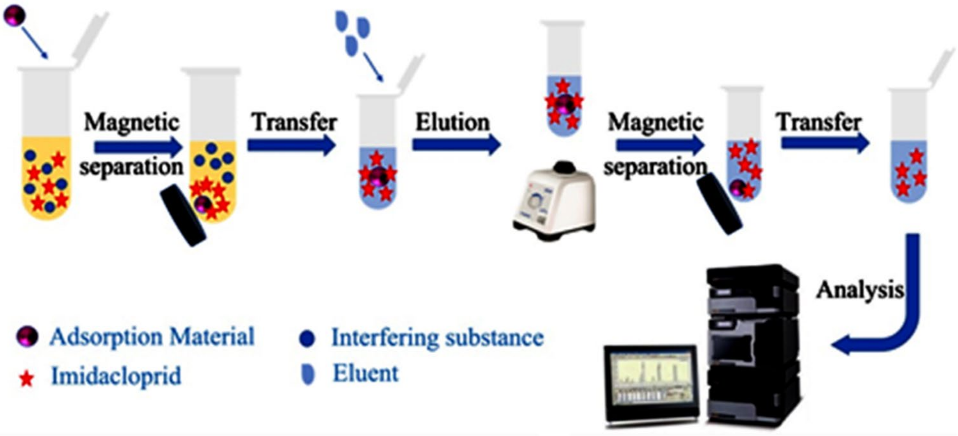

Fig. 2 Schematic illustration of adsorption and extraction procedure

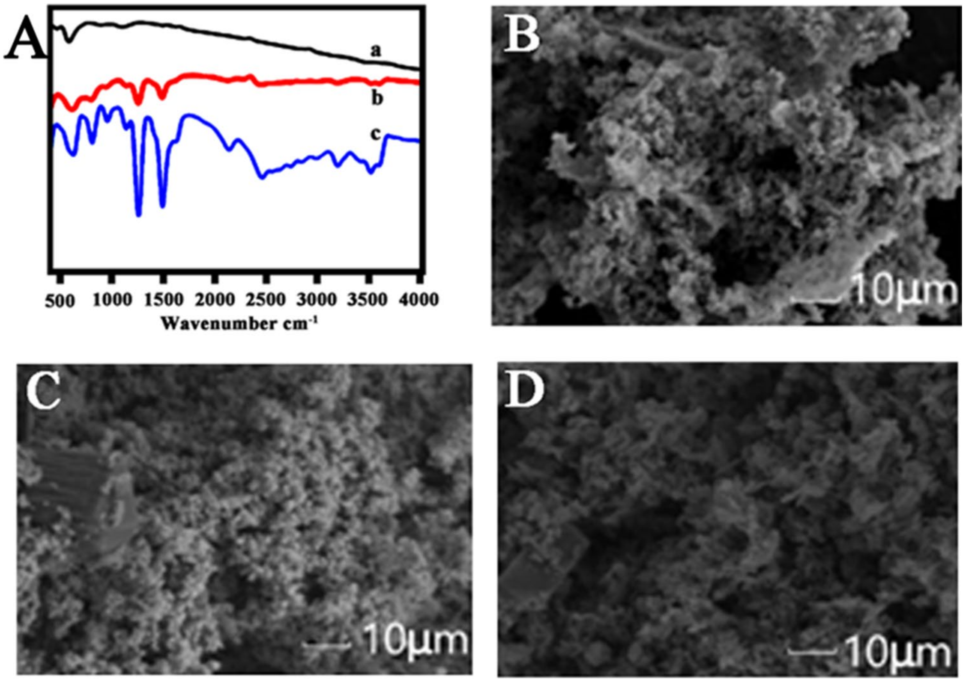

Fig. 3 FTIR spectra of Fe_3O_4 (**A**-a), MNIP (**A**-b) and MMIP (**A**-c) and SEM image of Fe_3O_4 (**B**), MNIP (**C**) and MMIP (**D**)

The cavities formed on the surface of MMIP can adsorb imidacloprid specifically because of the imitative structure of imidacloprid stemmed from the polymerization of dopamine. Meanwhile, the common properties of magnetic materials make the enrichment of imidacloprid easy from the water sample with a magnet (Fig. 4b).

The effect of the amount of MMIP on the adsorption of imidacloprid was optimized. For this, amounts of 1, 2, 4, 8, and 16 mg of MMIP were added into a standard solution of 10 mL containing 5 μg imidacloprid. Figure 5a shows that the adsorption quantity increased rapidly when the adsorption dosage increased from 1 to 4 mg. The adsorption quantity was increased slowly when absorbent dosage over 5 mg and gradually reached equilibrium. This result can be explained by the sufficient exposure of the adsorbent's adsorption sites in the solution when adsorbent dosage was low. Thus, the adsorption could reach saturation in a short time, and the adsorbent got a higher adsorption capacity. However, with the increasing adsorbent dosage, the number of unoccupied sorption sites on the adsorbent surface was excessive, but the fixed amount of the imidacloprid molecules in the solution (Wang et al. 2014).

Figure 5b shows peak areas abtained from chromatograms of imidacloprid solution treated according to the procedures desrcibred in "Extraction procedure" section. The value of the correlation coefficient (R^2) of the fitting line was 0.985, which indicate that the proposed method could be used to extract trace pesticide satisfied.

The effect of solution pH on the adsorption of MMIP to imidacloprid was investigated. The pH value of a solution can control an electrostatic interaction between the adsorbate and the adsorbent surface by inducing the charge distribution (Limchoowong et al. 2017). However, there were no significant differences in the adsorption efficiency in a pH range of 6.0–8.0, whereas there was just a little increase on adsorption quantity when pH is 6.5 (Fig. 5c).

The recognizing selectivity of MMIP was investigated by recoveries rate studies in distilled water between the target

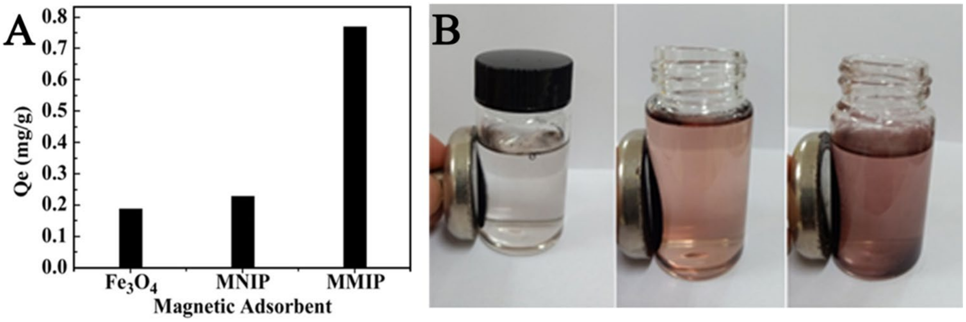

Fig. 4 The adsorption efficiency of Fe_3O_4, MNIP and MMIP (**a**), and images of solid–liquid separation under an external magnetic field (**b**)

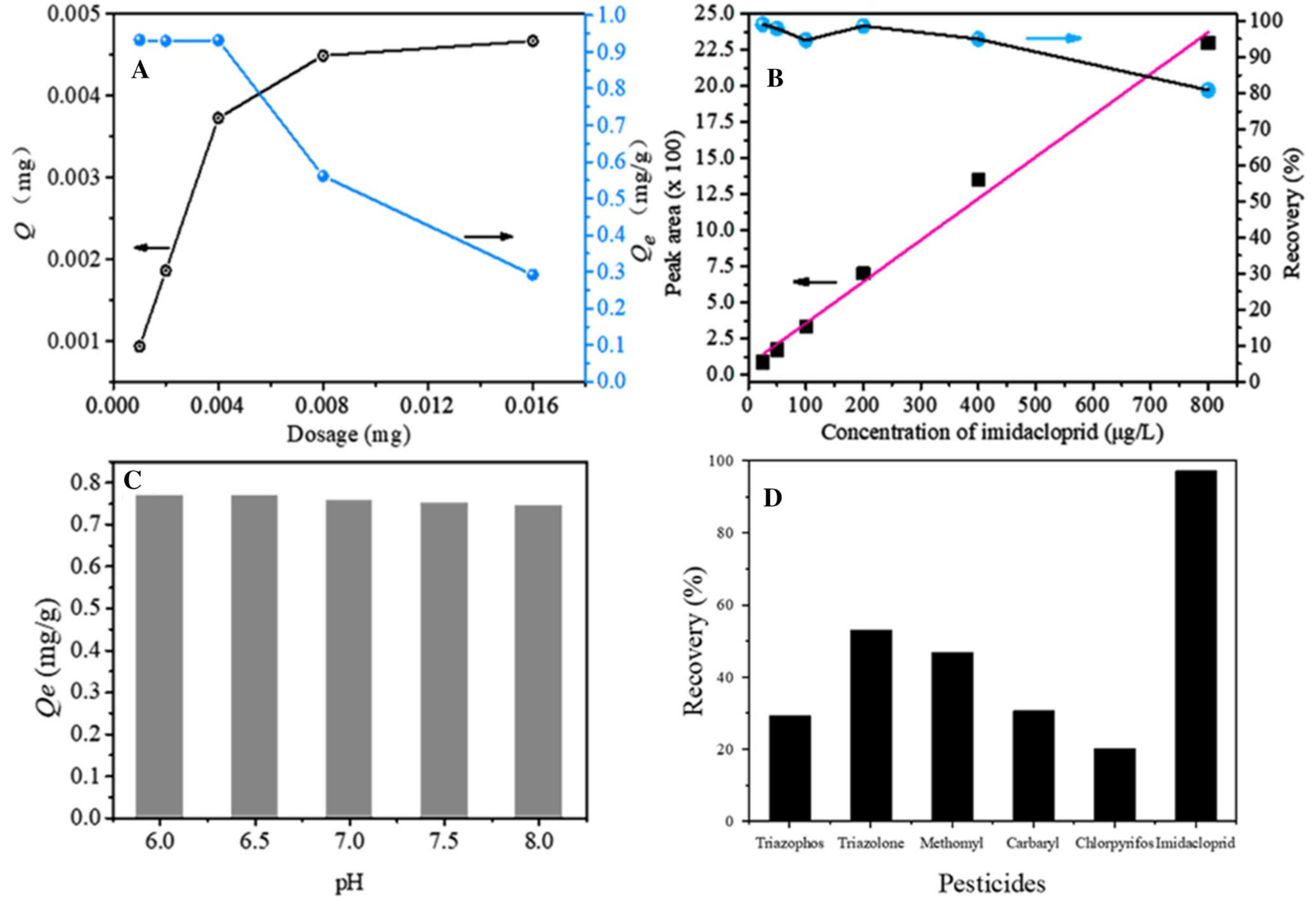

Fig. 5 Effect of adsorbent dosage on imidacloprid extraction by MMIP (**a**); the detectable chromatographic peak area of imidacloprid in eluent after extraction treatment by MMIP (**b**); effect of pH on imidacloprid adsorption using the MMIP as absorbent (**c**); the recognizing selectivity of MMIP to Imidacloprid against the analogs (**d**)

and analogs of the target, as shown in Fig. 5d. From the results, we found that the recoveries of the pesticides triazophos, triazolone, methomyl, carbaryl, and chlorpyrifos were lower than that of imidacloprid. It is indicated that the prepared MMIP extracts imidacloprid with a high affinity.

The mechanism of polymerization is shown in Fig. 6. The catechol groups of dopamine were firstly oxidized to the quinones thus formed dopaminequinone. Then dopaminequinone was transformed into leukodopaminechrome through nucleophilic reaction process. Leukodopaminechrome transferred into indolequinone from the further oxidation reaction. Polydopamine was formed under the inter molecules cross-linking reaction of indolequinone (Pan et al. 2009).

The high adsorption capacity and extraction efficiency of MMIP for imidacloprid could result in the miscellaneous adsorption mechanism synergistically. In the case of imidacloprid as template, polymerization of dopamine performed molecular imprinting program, so polymer has specific adsorption for imidacloprid. Meanwhile, there were plenty of negatively charged surfaces of the magnetic adsorbent due to the existence of numerous phenolic groups of polydopamine. These negatively charged surfaces could provide adsorption sites for electrostatic interaction or p–p stacking interaction with imidacloprid.

Analytical performance

To evaluate the applicability and accuracy of the propose analysis method based on imidacloprid extraction by MMIP and determination with HPLC, the adsorption and extraction of imidacloppridat trace levels were performed with the addition/recovery tests of imidacloprid in water samples. Table 1 shows that the determined imidacloprid concentrations were in a good agreement with the spiked concentrations values with the recoveries ranged from 88 to 97.4%, and the relative standard deviation (RSD) ranged from 1.2 to 3.3%. These results demonstrated the reliability of the method for the accurate determination of imidacloprid in real water samples.

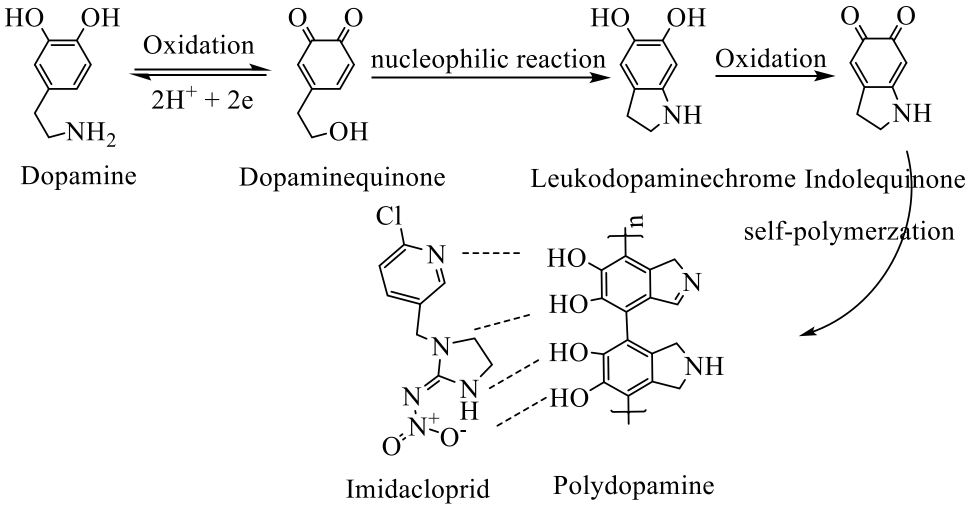

Fig. 6 The self-polymerization mechanism of dopamine

Table 1 Determination of imidacloprid in real water samples extracted using the MMIP and detected by HPLC–UV ($n=9$)

Sample	Spiked concentration (μg/L)	Found concentration (μg/L)	Recovery (%)	RSD (%)
S1	0.0	Not detect	–	–
S2	10	8.8	88.0	3.3
S3	50	48.7	97.4	1.2
S4	200	194.5	97.3	1.7

A comparative study was also evaluated between our proposed method and other reported methods for analysing imidacloprid, and the results are presented in Table 2. As can be seen, the LOD of the proposed method of this work (1 μg/L) for the imidacloprid were better than that of ion mobility spectrometry and comparable with the HPLC–MS in terms of LOD (Aria et al. 2019). The imidacloprid chromatogram is shown in Fig. 7. The recovery of imidacloprid extracted by proposed adsorbent was comparable with the recoveries by MIP made from monomer of methacrylic acid, acrylic acid, or p-vinylbenzoic acid (Kumar et al. 2018; Zhang et al. 2017; Jovanov et al. 2014). Moreover, the extraction procedure using MMIP will take place in a short time to adsorb the target. In addition, solid-phase was separated easily. Therefore, the proposed method is obviously featured with good effective, fast adsorption dynamics and easy-to-operate resulting from magnetism in the preconcentration and determination of imidacloprid.

Conclusions

The poly-dopamine functionalized magnetic particles were facilely prepared in one pot and used as magnetic dispersive solid-phase extraction adsorbents to simplify the isolation of imidacloprid from the water sample. The synthesized sorbent was characterized by SEM and FTIR. The prepared adsorbent MMIP exhibited good adsorption performance and selectivity for the imidacloprid. The adsorption saturation time of MMIP to imidacloprid was less than 30 min. The highly monodisperse and magnetically separable sorbent was applied for the isolation of imidacloprid with high efficiency due to the molecular imprinted polymer cavities on the surface of megnetic core. The adsorbed imidacloprid by MMIP was eluted using methonal followed by HPLC–UV analysis. The suggested method offered advantages such as simplicity, good enhancement factor in a short analysis time. As an outcome, the proposed method can be recommended as a proper alternative for the preconcentration imidacloprid in environmental matrix.

Table 2 Comparison of the proposed extraction method based on MMIP with other reported extraction methods based on solid adsorbent in the literature for the determination of imidacloprid

Detection method	Solid adsorbent	Sample	LOD	RSD (%)	Recovery (%)	References
Ion mobility spectrometry	An imidacloprid-selective MIP prepared using methacrylic acid as monomer, ethylene glycol dimethacrylate as cross-linker, acetonitrile as porogen, and azobisisobutyronitrilel as initiator	Chili and tomato plants	0.03 (μg/g)	11	102–104	Aria et al. (2019)
Liquid chromatography–tandem mass spectrometry (LC–MS/MS)	A MMIP prepared using nano Fe_3O_4 particles as magnetic cores, imidacloprid as template, acrylic acid as monomer, ethylene glycol dimethacrylate as cross-linker, and azobisisobutyronitrile as initiator	Honey and eggplant	0.05 (μg/g)	0.66 (eggplant) 2.06 (honey)	94.98 (eggplant) 94.15 (honey)	Kumar et al. (2018)
Electrochemical method with MIP/graphene modified glassy carbon electrode	MIP using *p*-vinylbenzoic acid as functional monomer layers on the surface of graphene sheets	Rice	0.10 (μM)	3.79	83.8	Zhang et al. (2017)
LC–MS/MS	Dispersive liquid–liquid microextraction	Honey	0.5–1.5 (μg/L)	3.21–10.20	69.2–113.4	Jovanov et al. (2014)
LC–MS/MS	Stainless-steel beads combined with solvent extraction	Citrus leaves	0.04 (μg/g)	8	92–102	Leiva et al. (2016)
High-performance liquid chromatography	A MIP prepared using methacrylic acid as the functional monomer, ethylene glycol dimethacrylate as cross-linking agent, acetonitrile as porogenic solvent	Water and soil	Not mentioned	Water (5.4) Soil (7)	Water (87.7–96.4) Soil (89.2–104.3)	Tang et al. (2009)
HPLC–UV	A MMIP prepared using Fe_3O_4 as core and dopamine as functional monomer	Water	1 (μg/L)	3.1	88–97.4	This work

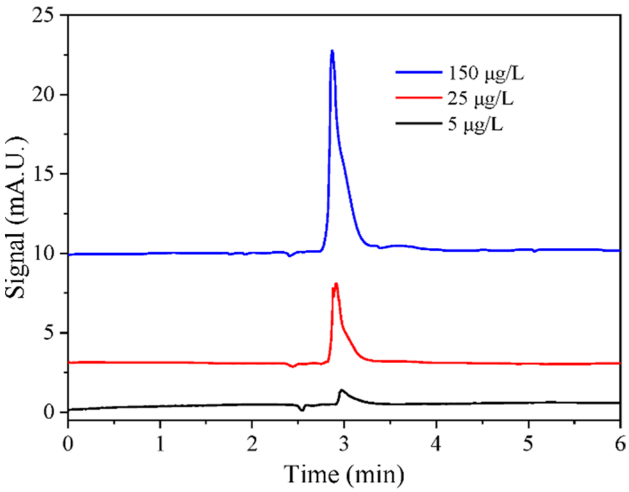

Fig. 7 The imidacloprid chromatogram in the different concentration

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