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Preparation of temperature sensitive molecularly imprinted polymer for solid-phase microextraction coatings on stainless steel fiber to measure ofloxacin

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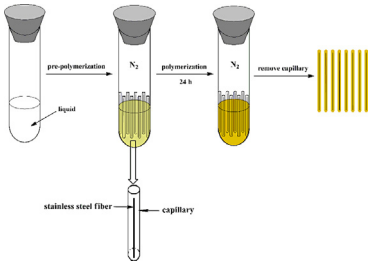
HIGHLIGHTS

- Dopamine was modified on stainless steel fiber as MIP fiber support.
- The stainless steel fiber was put into a capillary for the synthesis.
- Several capillaries were put together in same synthesis solution.
- The obtained MIP was evaluated well using different techniques.
- The MIP fiber was used to pretreat the milk samples to measure ofloxacin.

GRAPHICAL ABSTRACT



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ABSTRACT

A kind of new temperature sensitive molecularly imprinted polymer (MIP) with ofloxacin (OFL) as template was prepared for the coating of solid phase microextraction (SPME). Dopamine was self-polymerized on stainless steel fiber (SSF) as the SPME support followed by silanization. Then MIP was synthesized as SPME coating on the modified SSF in a capillary, with *N*-isopropyl acrylamide as temperature sensitive monomer and methacrylic acid as functional monomer. The synthesis could be well repeated with multiple capillaries putting in the same reaction solution. The obtained MIP fiber was evaluated in detail with different techniques and various adsorption experiments. At last the MIP fiber was used to extract the OFL in milk. Satisfied recoveries between 89.7 and 103.4% were obtained with the limit of quantification (LOQ_{LC}) of 0.04 μg mL^{-1} by the method of SPME coupled with high performance of liquid chromatography (HPLC).

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1. Introduction

The misuse of fluoroquinolone antibiotic drugs brought harm to poultry and human body, which caused wide attention of the society. For determination of the residual fluoroquinolone in complicated samples from environment, food and biology, suitable sample preparation could not be avoided [1–3].

Among the various sample preparation methods, miniaturization was welcomed because of the less reagent consumption. Solid phase microextraction (SPME) was one of economical methods used widely and its coating and support always were the focus of study. Mitani and Kataoka investigated a series of GC stationary phases – Carboxen 1006 (or 1010) PLOT, Supel-Q PLOT (Supelco), CP-sil 5 (or 19, or wax 52) CB as SPME coating to absorb the fluoroquinolone antibiotic drugs from water [4]. Molecularly imprinted polymer (MIP) was a competitive adsorbent in solid phase extraction (SPE) and SPME owing to the special recognition properties and the simple synthesis method. Kinds of MIPmaterials

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for determination of fluoroquinolone were prepared to be used as adsorbent for SPE [5–9], miniaturized SPE [10,11] and SPME [12,13]. Liu et al. [12] used MIP of polypyrrole/multi-walled carbon nanotubes (MIPPy/MWCNTs) composite coating on Pt fiber to carry out an electrochemically enhanced SPME for determination of fluoroquinolones in aqueous samples. Hu et al. [13] used fiber-in-tube SPME with MIPs as coating to determine the fluoroquinolones in animal-producing food samples. The methods set up in the above works got improved selectivity and sensitivity for the determination of tiny amount of fluoroquinolones in the samples.

Traditional SPME extraction was conducted by a thin film of coated materials on a solid support. Metal fibers, especial stainless steel fibers (SSF), became promising support substrates because of their inexpensive, firm and accessible properties. Different techniques have been developed for assembling the coatings on fibers such as chemical binding [14], electrochemical deposition [15], electrophoretical deposition [16], electroless plating [17] and sol–gel technology [18] besides the traditional physical deposition and adhesive methods. However, SSF was inert and it was necessary to improve the chemical activity before bonding the SPME coating [17].

Polydopamine was a versatile polymer as binding agents for coating various substrates [19]. The catechol and quinine functional groups on it could support a variety of reactions to create functional surfaces [20]. Using polydopamine as the initiator, it was successful on SSF to get hydroxyapatite [20] for SPME and poly(ethylene glycol) polymer [21].

In the work, we combined the factors of SSF, polydopamine and temperature sensitive polymer to obtain a new MIP material for the determination of ofloxacin in milk.

2. Experimental

2.1. Materials

Ofloxacin (OFL), enrofloxacin (ENR), ciprofloxacin (CIP) and sulfamerazine (SMZ) were purchased from Shanghai source biological technology Co., Ltd. (Shanghai, China). The compound structures are shown in Fig. 1. 3-Methacryloxypropyltrimethoxysilane (3-MPS), *N*-isopropyl acrylamide (NIPAAm), dopamine, methacrylic acid (MAA), acrylamide (AM) and ethylene glycol dimethacrylate (EGDMA) were purchased from Aladdin reagent Co., Ltd. (Shanghai, China). MAA and EGDMA were used after vacuum distillation. Azodiisobutyronitrile (AIBN) was purchased from Tianjin Yuefeng chemical Co., Ltd. (Tianjin, China) and used after recrystallization. Methanol (MeOH), ethanol (EtOH), acetone, dimethyl sulfoxide (DMSO), chloroform, acetonitrile (ACN), acetic acid (HAc) and others were purchased from Tianjin Guangfu chemical Co., Ltd. (Tianjin, China). All the reagents were of analytical grade. Milk samples were bought in a local supermarket (Lanzhou, China). SSF (400 μm O.D.) was purchased from a hardware Company (China). Quartz capillary (1 mm I.D.) was purchased from Huaxi medical university instrument plant (Chengdu, China).

2.2. Instrument and chromatographic conditions

UV–vis experiments were carried out on a UV–vis spectrophotometer (TU-1810, PUXI, Beijing); the Fourier transform infrared (FTIR) spectra were acquired with a Nicolet 20 NEXUS 670 FTIR spectrophotometer (Ramsey, MA, USA) using KBr pellets; scanning electron microscope (SEM) images of materials were obtained using an S-94 4800 SEM (Hitachi, Japan). Nitrogen adsorption–desorption measurements were performed at 77 K on a Tristar ASAP 2020M Surface Area and Porosimetry analyzer (Micromeritics Instrument Corp., USA). The surface-area measurement was based on the Brunauer–Emmett–Teller (BET) method, and the pore-size distribution was based on the Barrett–Joyner–Halenda (BJH) formula. Ultrapure water was from MILLI-Q ultrapure water system (Millipore, Bedford, MA, USA).

Chromatographic analysis was carried out on a high performance liquid chromatograph (HPLC) system (Varian, USA) equipped with two 210 pumps, 325 UV–vis detector, Varian star

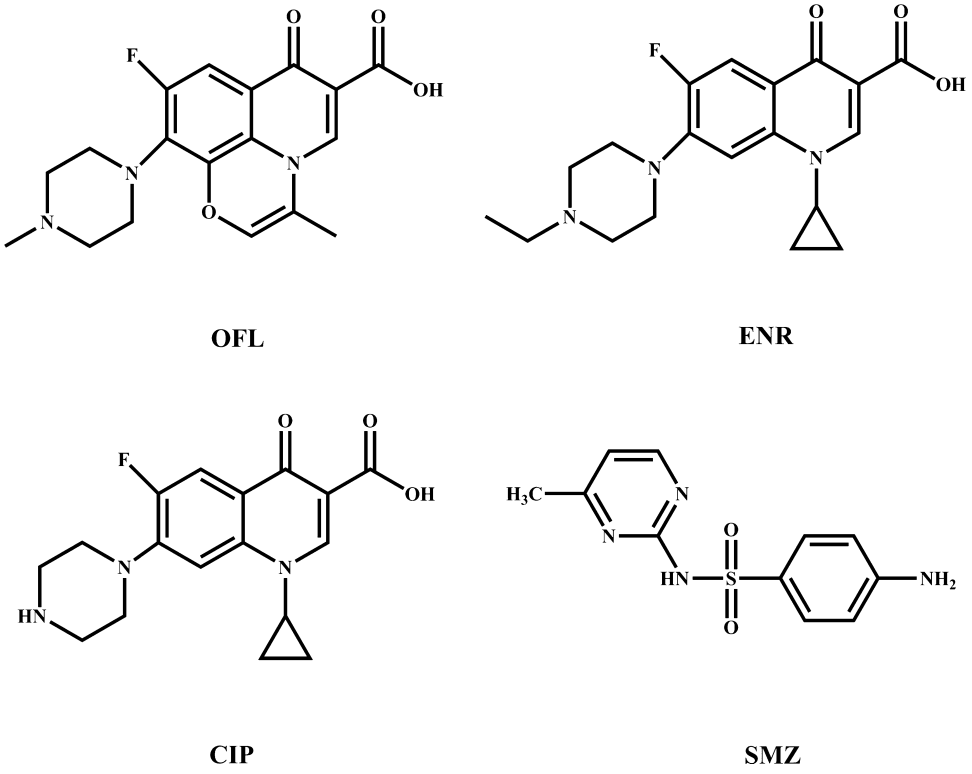

Fig. 1. Structures of compounds studied in the work.

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