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Metal organic framework derived Zn/N co-doped hydrophilic porous carbon for efficient solid phase microextraction of polar phenols

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Abstract

MOF-derived zink and nitrogen co-doped porous carbon (ZNPC) was synthesized through the pyrolysis of MOF-5-NH$_2$ and used as a solid-phase microextraction (SPME) coating material. Coupled with gas chromatography-mass spectrometry (GC–MS), headspace SPME (HS-SPME) based on ZNPC was adopted for the determination of phenols in food samples. The co-existence of N and Zn in ZNPC endows the derived carbon superior hydrophilicity, which is highly beneficial for phenols capture. After optimizing the conditions of extraction and desorption, a sensitive analytical method was established with low limits of detections (LODs, 0.73–2.3 ng g^{-1}) and wide linear ranges (5–5000 ng g^{-1}). Both the intra-fiber repeatability (RSDs from 2.8–7.3%) and inter-fiber reproducibility (RSDs from 9.7–11.7%) were satisfactory. The established method was applied to phenol determination in beef jerky and duck neck with satisfactory recoveries of 81.2–120.4% and RSDs of 2.8–9.9%, which met well with the requirement of practical application.

Keywords Solid-phase microextraction · Porous carbon · MOF-derived materials · Phenols · Hydrophobic sorbent

Introduction

Phenols, one of the most frequently detected contaminants, would result in chronic intoxication even at low concentrations due to the accumulation and even generate acute poisoning and death at high concentrations [1]. Several phenolic compounds have been identified as priority organic contaminants by the US Environmental Protection Agency (EPA), where the maximum allowable concentration of single phenol in water is 0.1 μg L^{-1} [2]. To guarantee food safety, it is of great importance to develop environmentally, fast, and accurate analytical methods for the determination of trace phenols [3]. However, the sensitivity analysis is challenging due to the complex matrix interference and trace concentration [4].

Currently, several advanced sample preparation techniques including magnetic solid-phase extraction (MSPE), liquid–liquid partition extraction (LLPE), and solid-phase microextraction (SPME) have been adopted to eliminate the interference of matrix and realize the enrichment of the target. Among them, SPME is a competitive and powerful technique that integrates separation, enrichment, and sample injection into one step [5, 6]. Impressively, SPME has been widely applied in the preconcentration of various compounds from environment [7], foods [8], metabolomics [9], and other fields [10].

Since the properties of the coating have a significant impact on the application range, selectivity, and sensitivity of the established analytical method [11, 12], the development of new inexpensive coating materials with stable and excellent extraction performance has always been the emphasis of research. Porous carbons have attracted extensive attention owing to their easy preparation, high specific surface area, and tailored structure [13, 14]. For example, Zheng et al. [15] prepared a polydopamine-modified ordered mesoporous carbon as SPME coating. Combined with GC–MS, the developed method exhibited low limits of detection (LODs, 0.08–0.38 ng L^{-1}) towards various phenols. However, in general, porous carbons are hydrophobic, which are unfavorable for the capture of polar phenols.

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Therefore, it is valuable to develop hydrophilic porous carbons with simple synthetic methods to capture polar phenols efficiently.

Recently, metal–organic frameworks (MOFs) have been utilized as ideal precursors to prepare porous carbons through a facile and single-step calcination procedure due to their diverse structure and high porosity [16, 17]. Several studies have reported that the MOF-derived porous carbons exhibited excellent extraction performance as SPME coating due to their fantastic characteristics [18–20]. For example, Ni et al. [20] prepared the Zr and N co-doped hydrophilic porous carbon using UiO-66-NH$_2$ as precursor, which was used as an efficient SPME coating to extract trace phenols from water. The study showed that porous carbon integrating both metal component and heteroatom elements was helpful to adjust the hydrophobicity, which is beneficial for polar phenols capture. Thus, the facile preparation and superior capture performance make it attractive to fabricate hydrophilic carbons by taking MOFs as precursors [21]. Nevertheless, the influence of metal component and heteroatom co-doped on the hydrophilicity/hydrophobicity of the resulting porous carbons and their extraction performance has not yet been fully noticed.

In this research, MOF-5-NH$_2$, one of the most commonly used MOFs, was selected as a precursor to prepare Zn and N co-doped porous carbon (ZNPC) by direct carbonization. As a proof of concept, the obtained ZNPC featuring good hydrophilicity was used as SPME coating coupled with gas chromatography-mass spectrometry (GC–MS) to determine trace phenols in food products. Extraction performance using the materials with only Zn or N doped was also evaluated to study the influence of element doping on the hydrophilicity and extraction behavior.

Experimental

Chemicals and reagents

Zinc acetate dihydrate, 2-aminoterephthalic acid, terephthalic acid, N, N-dimethylacetamide (DMAc), hydrochloric acid, sodium hydroxide, sodium chloride, and methanol were purchased from Sinopharm Chemistry Reagent Co. Ltd. (Shanghai, China). Phenols standard solution containing 2,4,6-trichlorophenol (2,4,6-TCP), 2-nitrophenol (2-NP), 2,4-dichlorophenol (2,4-DCP), 2,4-dimethylphenol (2,4-DMP), and 2,6-dimethylphenol (2,6-DMP) were obtained from Accustandard (United States). Stainless steel wire (SSW, φ 0.14 mm) was got from Shenzhen Bao Fang Metal Materials (Guangdong, China). Commercial polyacrylate (PA, 85-µm thickness) fiber was purchased from Supelco

(Bellefonte, PA, United States). Sylgard 184 silicone gel was provided by DOW CORNING (United States).

Apparatus

The SPME procedure was completed on a multipurpose sampler (MPS, GERSTEL, Germany). The analysis of phenols was performed on a GC–MS (QP2010 PLUS) equipped with an Rtx-Wax capillary column (30 m × 0.25 mm × 0.25 µm) (SHIMADZU, Japan). The conditions of the column were described as follows: initially, the temperature was held at 100 °C for 1 min before heat up to 120 °C within 4 min. Subsequently, the temperature was raised to 150 °C within 3 min and kept for 1 min, finally, raising the temperature to 230 °C and hold for 2 min. The samples were desorbed at 230 °C in the injector, which was operated in splitless mode. The carrier gas was high-purity helium (99.999%) and the flow rate was maintained at a constant flow rate of 1.5 mL min^{-1}. The operating conditions of MS are summarized as follows: the temperature of both the transfer line and ion source is kept at 220 °C. Besides, the ion source was operated in electron impact mode (EI) (70 eV) and the detection was conducted in SIM mode. The specific parameters of the SIM mode are shown in Table S1.

N$_2$ adsorption − desorption isotherms of the as-prepared materials were measured after degassing at 150 °C for 12 h. The Brunauer − Emmett − Teller (BET) method was utilized to estimate the specific surface areas. Scanning electron microscopy (SEM, HITACHI, SU8010, Japan) and transmission electron microscopy (TEM, JEM-2010 electron microscope, Japan) were adopted to gain insight into the morphology of ZNPC. X-ray diffractometer (XRD, D8 Advance, Bruker, Germany) with Cu-Kα radiation and Labor Raman HR (Horiba, USA) were employed to study the structural information. Fourier transform infrared (FT-IR) spectra were performed on a Nicolet 6700 (Thermo Fisher, USA). The elemental distribution images were acquired through energy-dispersive X-ray spectroscopy (EDX). The water contact angle was measured using a Drop Shape analyzer (DSA100; Krüss, Hamburg).

Fabrication of the SPME fiber

The synthesis of MOF-5-NH$_2$ and MOF-5 was referred to the literature with a slight modification [22]. The detailed information was presented in the Electronic Supporting Material (ESM). To prepare ZNPC, a porcelain boat loaded with 1.0 g MOF-5-NH$_2$ was put in the center of a tubular furnace. Then, the temperature was raised from room temperature to 800 °C at a heating rate of 10 °C min^{-1} under N$_2$ atmosphere and maintained for 1 h. After cooling to room temperature, the resulting product, denoted as ZNPC, was collected. Similarly, ZPC was

prepared through the same procedure by taking MOF-5 as a precursor. NPC was obtained by treating ZNPC with 10% hydrochloric acid to remove element Zn.

Clean SSW and silicone glue were prepared according to the previous literature [23]. After taking out from the configured adhesive, the treated SSW was rolled in the ZNPC powder, resulting a uniform coating. Subsequently, the ZNPC-coated fiber was conditioned at 150 °C for 15 min. The aforementioned procedure was performed 3 times in succession. Finally, the homemade fiber was aged for 2 h at 250 °C under N_2 atmosphere.

Headspace SPME (HS-SPME)

The solution (1 μg mL^{-1}) containing 5 phenols was obtained through diluting the standard solution (1000 μg mL^{-1}) with methanol. Phenols sample solution (1 ng mL^{-1}) was prepared by diluting the phenols stock solution with ultra-pure water. Afterwards, the homemade coating was inserted into the headspace of a vial containing 10 mL phenols solution under the assistance of MPS. After extraction, the coating was transferred into the inlet of GC–MS for thermal desorption and subsequent analysis.

Preparation of food sample

Meat samples (beef jerky and duck neck) were purchased from the local supermarket (Wuhan, China). The sample preparation procedure was as followed: firstly, the meat samples were freeze-dried and ground into powder. Then, 0.1-g sample powder was dispersed in 1 mL dichloromethane:n-hexane (V/V = 2:1) for 10 min under the ultrasonic treatment. Extraction solvent was filtered by the 0.45-μm organic filter as a reserve solution. Then, 100 μL of the reserve solution was added into 10 mL saturated NaCl solution (pH 4) and the sample was analyzed under optimum conditions.

Results and discussions

Characterizations of the coating material

The morphology of ZNPC coating and ZNPC powder was characterized by SEM and TEM. Figure 1a and Fig. 1b show that ZNPC particles have a rough surface. Figure 1c shows the surface of the SSW is smooth with a diameter of 147 μm. Figure 1d shows that the surface of SSW was uniformly coated by ZNPC and the diameter of the ZNPC coated fiber is 218 μm. As a consequence, the thickness of

Fig. 1 **a** SEM and **b** TEM images of ZNPC powder, SEM images of **c** bare SSW and **d** ZNPC-coated fiber.
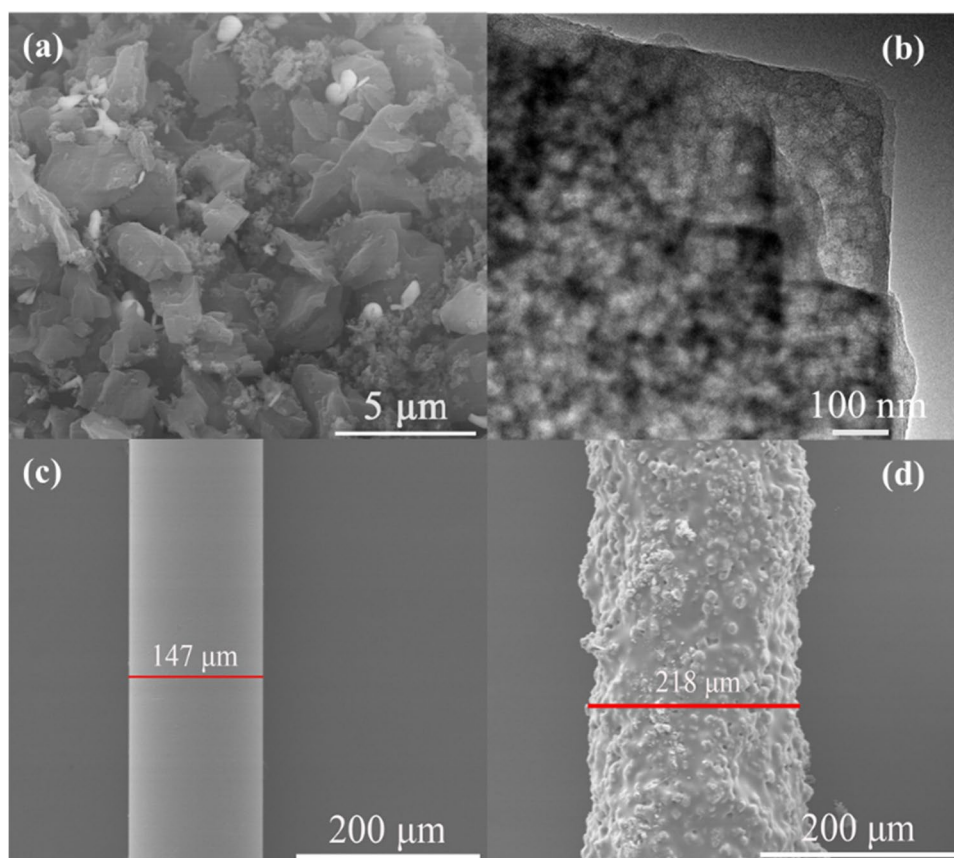
the ZNPC coating is calculated to be ~ 35 μm. The uneven surface endows the ZNPC coating with ample accessible binding sites for phenols capture.

As shown in Fig. S1, the XRD patterns of MOF-5-NH$_2$ are consistent with these reported in the literature [24], demonstrating that it was successfully synthesized. In Fig. 2 a, the patter observed at 31.9°, 34.6°, 36.5°, 47.7°, 56.7°, 63.2°, 68.2°, and 69.2° of ZNPC and ZPC can be ascribed to (100), (002), (101), (102), (110), (103), (112), and (201) crystal planes related to hexagonal zinc oxide (ZnO, JCPDS 01–1136), respectively. These results suggest that the zinc clusters in MOFs have converted into ZnO nanoparticles during the carbonization procedure [25]. After treating with hydrochloric acid, the XRD patterns corresponding to ZnO disappeared, suggesting the ZnO nanoparticles have been completely removed. As shown in Fig. 2b, peaks at 1639 cm^{-1} corresponding to the C = C vibration derived from benzene skeleton of the porous carbons are observed, indicating the possibility of formation π-π stacking between MOF-derived carbons and aromatic hydrocarbons. The peak at 3449 cm^{-1} can be related to the stretching vibration

of -OH. Furthermore, the absorption peak observed at 1560 cm^{-1} can be ascribed to C = N stretching vibration. The presence of Zn is conducive to the retention of N, and the absorption peak at 1415 cm^{-1} was caused by -OH flexural vibration.

Fig. 2c shows the N$_2$ adsorption–desorption isotherms of ZNPC, ZPC, and NPC, and the specific surface areas of which are 440.2 m^2 g^{-1}, 332.4 m^2 g^{-1}, and 784.8 m^2 g^{-1}, respectively. Compared with that of ZNPC, the specific surface area of NPC showed a significant increase due to the removal of ZnO nanoparticles, yielding additional pores. The adsorption–desorption isotherms are type IV, which represents large numbers of mesopores and some macropores in the material [26]. Figure 2d shows that the I_D/I_G of the carbon materials were all close to 1, indicating the existent of a high graphitization degree, possibly because of the catalytic graphitization in the presence of Zn [27–29].

As shown in Fig. 3a and 3b, ZPC and NPC are hydrophobic carbon, of which water contact angles are 146° and 123°, respectively. Interestingly, the co-doping of Zn and N changes the hydrophobic carbon into hydrophilic carbon,
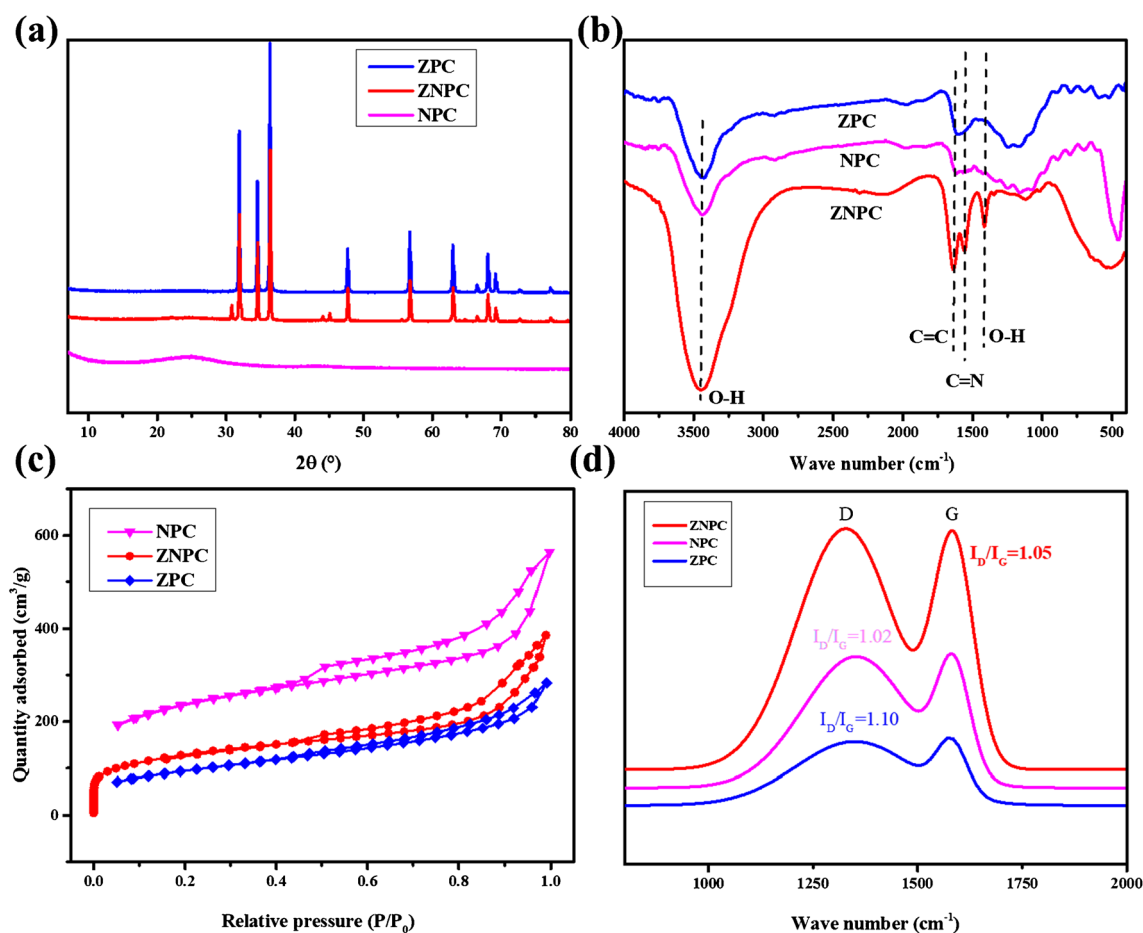

Fig. 2 **a** XRD patterns; **b** FT-IR spectra; **c** N$_2$ adsorption–desorption isotherms; and **d** Raman spectra of ZNPC, NPC, and ZPC.
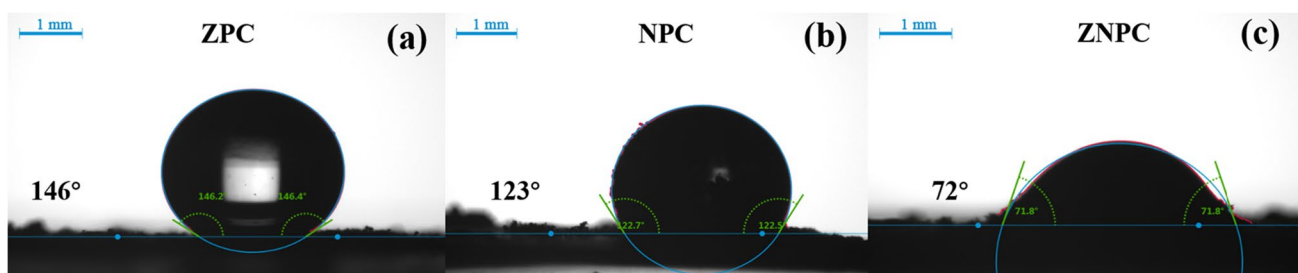
Fig. 3 The water contact angle of **a** ZPC, **b** NPC, and **c** ZNPC

where the water contact angle of ZNPC is 72° (Fig. 3c) The high hydrophilicity of ZNPC might be contributed by the synergistic effects between the doped nitrogen and ZnO nanoparticles embedded in porous carbon, both of which exhibit polar nature. The hydrophilic characteristic of ZNPC may enhance the capture of phenols via the formation of strong hydrogen bonding [20]. The images of element mapping (Fig. S2) show that C, N, O, and Zn are distributed evenly, providing accessible binding sites for phenols capture. All these results demonstrated the successful preparation of ZNPC.

Optimizations of parameters of SPME

To achieve the best extraction efficiency, the relevant parameters include (A) extraction temperature; (B) extraction time; (C) desorption temperature; (D) desorption time were optimized (ESM). The experimental results (Fig. S3) demonstrate that the optimal extraction and desorption conditions for ZNPC coating are as follows: extraction temperature,
70 °C; extraction time, 50 min; desorption temperature, 230 °C; and desorption time, 2 min.

Comparison in analytical performance with commercial PDMS fibers

To further understand the extraction performance of the ZNPC coating, the as-developed method was compared with that based on the commercial PA coating (85 μm). As shown in Fig. 4 a, compared with NPC, ZPC, and commercial PA coating, ZNPC shows the best extraction performance. The results might be attributed to these factors: firstly, the formation of strong hydrogen bond interaction between hydrophilic ZNPC and phenols; secondly, larger specific surface area can provide more available binding sites for the uptake of phenols; thirdly, the π-π stacking interaction is also conducive to the adsorption. The property of the coating for repeated use was also investigated. As shown in Fig. 4 b, even after 90 cycles, the extraction efficiency of the ZNPC coating only showed a slight change. As shown in Fig. S4, the morphology of ZNPC coating fiber after 90 cycles also
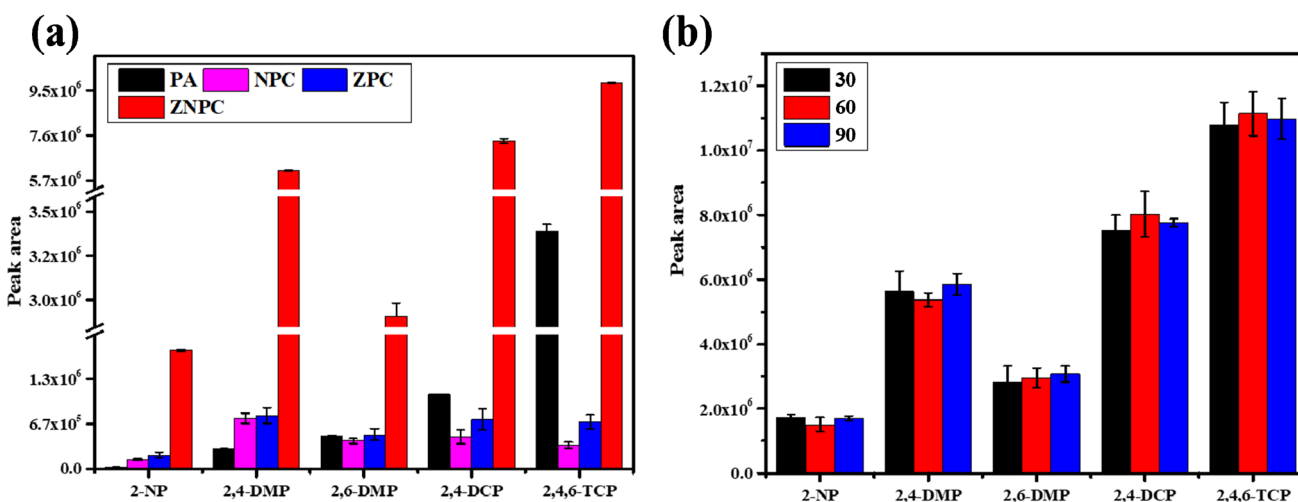

Fig. 4 **a** Comparison of extraction performance by using ZNPC, ZPC, NPC, and PA (thickness: 85 μm). **b** The extraction performance of the ZNPC coated SPME fiber under different cycles. Extrac-
tion conditions: extraction time of 50 min, extraction temperature of 70 °C, salt concentration of 37% (NaCl, wt.%), desorption time of 2 min, and desorption temperature of 230 °C.

showed negligible change in comparison to the initial one, suggesting the ZNPC coating has an excellent stability.

Evaluate the applicability of the procedure

Under optimized conditions, the performance of ZNPC coating for the extraction of phenols was evaluated. As depicted in Table S2, the established method showed an excellent linearity, high coefficient of determination (R^2), and low LODs. The RSDs of five phenols were 2.8~7.3% and 9.7~11.7% for single fiber and fiber-to-fiber, respectively. Besides, the enrichment factors (EFs, calculated according to the ratio of the sensitivity of target after extraction to that of before extraction, i.e., $EFs = S_{after}/S_{before}$) of the five phenols were in the range of 14,978 to 59,980.

To testify the application potential of ZNPC coating on actual samples, the proposed method was used to monitor the phenols in different food samples, beef jerky, and spicy duck neck. The chromatograms of real sample (beef jerky) before and after spiking (10 ng g^{-1}) were shown in Fig. S5.

According to the results shown in Table 1, only 2,4,6-TCP was detected in different meat samples. Satisfactory results were obtained for all the spiked samples, where the recoveries and RSDs ranged from 81.2–120.4% and 2.8–9.9%, respectively. All these results indicated that the developed method is feasible for trace phenols analysis in beef jerky and duck neck. As displayed in Table 2, the proposed method exhibited comparable or even better results in comparison to other methods, demonstrating that ZNPC is an intriguing adsorbent for the extraction of polar phenols in real samples with a complex matrix. Despite its distinct advantages, there are still some limitations. Firstly, since silicone glue was employed to ensure the adherence of ZNPC onto the fiber, the synthetic conditions involving during the coating preparation procedure should be precisely controlled to ensure reproducibility and avoid pore blocking. In addition, due to its large specific surface area and inherent carbon skeleton, ZNPC also exhibited a certain adsorption capacity towards other organic compounds such as weakly polar benzene series (BTEX) and nonpolar polycyclic aromatic

Table 1 The analytical results of real samples

Sample	Compounds	Measured concentration (ng g^{-1})	Low spiked concentration (10 ng g^{-1})		High spiked concentration (50 ng g^{-1})	
			Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
Beef jerky	2-NP	ND	109.8	5.1	103.7	4.0
	2,4-DMP	ND	100.9	3.5	92.6	3.0
	2,6-DMP	ND	97.8	2.8	98.9	3.1
	2,4-DCP	ND	114.9	6.8	120.4	5.0
	2,4,6-TCP	5.1	100.8	3.8	110.1	4.1
Spicy duck neck	2-NP	ND	83.3	9.9	96.0	5.4
	2,4-DMP	ND	81.2	8.0	87.5	4.0
	2,6-DMP	ND	114.3	9.5	109.6	7.3
	2,4-DCP	ND	105.4	6.1	107.4	6.9
	2,4,6-TCP	5.4	94.1	4.9	110.9	4.5

ND, not detected.

Table 2 Comparison of the performances of the proposed method with other published procedures for the determination of phenols

Analytical method	Materials	Sample	Linear range (ng L^{-1})	LODs[a] (ng L^{-1})	RSD (%)	Life time (times)	Refs
SPME/GC–MS	OMP-NH$_2$	Pond & river water	0.2–10,000	0.05–0.16	4.0–6.1	150	[8]
SPME/GC–MS	Fe$_3$O$_4$@HPS	Water and sediments	10–300	1.0–3.1	-	-	[30]
SPME/GC–MS	MOF-177	Surface, river & waste water	100–50,000	15–43	2.8–5.5	-	[31]
SPME/GC–MS	Co-MONT-	Waste water	0.5–1000	0.07–0.18	4.3–8.4	100	[32]
SPME/GC–MS	SNW-1	Honey	0.1–100	0.06–0.2 ng g^{-1}	4.3–9.7	110	[33]
SPME/GC–MS	GO/POE	Pearl river water	5–1000	0.12–1.36	1.5–4.4	100	[34]
SPME/GC–MS/MS	PPc-CMPs	Tap, snow, and waste water	0.1–1000	0.02–0.05	2.5–8.1	100	[35]
SPME/GC–MS/MS	SCSs-COOH	Tap, river, and lake water	1–1000	0.26–2.63	2.0–9.0	-	[36]
SPME/GC–MS	ZNPC	Beef jerky & spicy duck neck	5/10–5000 ng g^{-1}	0.73–2.3 ng g^{-1}	2.8–9.9	90	This work

[a]LODs were calculated based on the signal-to-noise ratio of 3 (S/N = 3).

hydrocarbons (PAHs) through π-π stacking interaction, affecting the extraction selectivity (Fig. S6).

Conclusions

In conclusion, ZNPC was prepared through direct carbonization by utilizing MOF-5-NH$_2$ as the precursor and utilized as potential SPME coating for the efficient extraction of phenols. Compared with that of NPC and ZPC, ZNPC featuring excellent thermal stability, good hydrophilicity (water contact angle was 72°), and large specific surface area (440.2 m^2 g^{-1}) exhibited much better extraction performance, benefiting from the co-doped of N and Zn. Coupling with GC–MS, a sensitive and reliable analytical method for the analysis of trace phenols in food products was established. To further improve the extraction performance, the development of a facile method to prepare such coating by growing nitrogen-containing MOF material on the metal wire substrate through in situ synthesis and subsequently carbonization is of more interest. Furthermore, the application of metal and heteroatom co-doped porous carbon for other contaminants will be explored in the future.

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Declarations

Competing interests The authors declare no competing interest.

Credit authorship statement Qian Yan: investigation, software, and writing—original draft; Lijin Huang: conception, review and editing, and funding acquisition; Weikang Guo: data analysis and review; Lei Ouyang: review and editing; Qin Shuai: resource acquisition.

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