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*Review Article*

# Trends of Novel Functional Nanomaterials for Analysis of Sulfonamide Residues in Food Products

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The residues of sulfonamides (SAs) may transfer to the human body through the food chain, causing risks to human health. Therefore, it is of significance to address the SA residues in food products. Recently, the development of nanomaterial adsorbents with fast adsorption, low toxicity, robustness, and reusability has attracted much attention. CiteSpace software was used in this work to analyze studies on the detection of SAs in the past five years. Furthermore, novel nanomaterials, including magnetic nanomaterials, metal–organic framework materials (MOFs), and covalent organic framework materials (COFs), are reviewed in detail for extraction and analysis of SAs in food, showing the advantages in the adsorption and analysis performance of these nanomaterials. Challenges and prospects of related research are also discussed to clarify the future directions for the achievement of food safety. This review is expected to provide new insights for the development of efficient adsorbents and analytical methods for food pollutants with potential applications.

## 1. Introduction

Sulfonamides (SAs) are a class of synthetic antibacterial drugs with a p-aminobenzenesulfonamide structure. They are widely used to prevent and treat bacterial infectious diseases in animals because of their wide variety, broad antibacterial spectrum, stable nature, convenient use, good sterilization effect, and other characteristics. However, SAs may transfer to the human body through the food chain, causing risks to human health [1]. For example, long-term intake may cause damage to urinary system and liver function, inhibition of normal gastrointestinal tract flora, anemia, allergic reaction, bacterial drug resistance, and other adverse reactions [2]. Therefore, the problem of SA residues above the maximum residue limits (MRL) in food has attracted increasing attention.

Highly sensitive and selective technologies for the separation and analysis of SAs have been developed rapidly with the progress in nanomaterials. Advanced sample pretreatment technology is of great significance and value for accurate detection to ensure food safety. The development of nanomaterial adsorbents with fast adsorption, low toxicity, robustness, and reusability has attracted much attention [3, 4]. In recent years, new porous nano/micromaterials, such as magnetic functional adsorption nanomaterials, metal–organic framework materials (MOFs), covalent organic framework materials (COFs), or mixed nanomaterials, have an important role as functional materials in food safety inspection, especially in the research of complex food sample matrices [5–7].

In this work, CiteSpace software was used to analyze studies on the detection of sulfonamide antibiotic residues in the past five years. The literature on the detection of SAs mainly focused on chromatographic analysis, mass spectrometry analysis, and other related fields. Novel nanomaterials, including magnetic nanomaterials, MOFs, and COFs, for extraction and analysis of SAs in food, are reviewed. Challenges and prospects of related research are also discussed. This review is expected to provide a new perspective for the development of efficient adsorbents and analytical methods for food contaminants, including SAs.

## 2. Literature Analysis Based on CiteSpace

Food safety problems potentially caused by the SA residues have attracted extensive attention. Scholars have devoted themselves to relevant research on the detection and analysis of SAs in food and achieved remarkable outputs. On November 1, 2022, 643 valid studies have been obtained after deduplication by CiteSpace based on the core database in WoS launched by the American Institute of Science and Technology Information (ISI), taking SAs AND food or meat or fish or milk AND detection or analysis or determination as the theme, 2018-2022 as the publication year, article as the literature type for retrieval [8]. The visiting institution is Jiangsu University of Science and Technology. A visual atlas of the 605 articles is drawn based on CiteSpace 6.1.R3 software, which mainly analyzes the cocitation of keywords and literature. The time range is set from 2018 to 2022, and the time slice is one year. Each time slice selects the first 50 nodes (TopN = 50) to draw the atlas.

Figure 1 for the keyword cooccurrence atlas shows the number of nodes on the upper left ($N = 281$). The number of each node represents a keyword. The size of the node represents the number of documents in which the keyword appears. The different colors of the tree ring represent the year of publication of the corresponding article on the lower left. The top 10 frequently appearing keywords are SAs, milk, residues, antibiotics, solid phase extraction (SPE), high-performance liquid chromatography (HPLC), food, drug resistance, validation, and liquid chromatography. As shown, these keywords are the research focus in the past five years. Antibiotics and drug resistance are also the hotspots that have received wide attention. Milk is the most mentioned food matrix. SPE, HPLC, and liquid chromatography are the important current techniques for SA analysis with promising applications.

CiteSpace is applied for keyword clustering of cited literatures to obtain a visual atlas of research frontiers (Figure 2). In the upper left corner, it found the cluster $Q = 0.5959$ and $S = 0.8664$, indicating that the results are highly reliable, and there is a total of $N = 317$ literature in the figure, which constitutes an association node. The nine research frontiers are # 0 sulfonamide, # 1 urea, # 2 liquid chromatography, # 3 mass spectrometry, # 4 whole genome sequencing, # 5 magnetic solid phase extraction (MSPE), # 6 dihydroxyvalerate synthetase, # 7 fluorescence, and # 8 anaerobic digestion. It can be seen from the analysis results of CiteSpace that the detection of SAs mainly focuses on chromatographic analysis, mass spectrometry, fluorescence, and other related fields. SPE provides a good pretreatment method for the separation and analysis of SAs. MSPE combined with dispersed liquid-liquid microextraction (DLLME) proposes better purification ability than single MSPE.

With the progress in nanomaterials, highly sensitive and selective technologies for separating and analyzing SAs have been developed rapidly as alternatives in real food samples [9]. However, the current techniques obtained by CiteSpace are lacking with nanomaterial-related content, while the novel nanomaterials should be highlighted to show their unique advantages and applications for SA analysis in food products.

## 3. Separation and Analysis of SAs by Using Nanomaterials

Nanomaterials and nanocomposites presented the advantages to enrich the SAs in food products showing a low detection limit, good linear relationship, and satisfied recovery [10, 11]. Advanced sample pretreatment technology combined with nano/microfunctional materials as adsorbent (Table 1) has great significance and value for accurate detection to ensure food safety. Scholars have focused on the development of functional nanomaterial adsorbents that are easy to prepare and have fast adsorption and separation performance, low toxicity, robustness, and reusability [12]. New porous nano/micromaterials play an important role as functional materials in food safety inspection, especially in research of complex food sample matrices [13].

Magnetic nanomaterials have the advantages of simple separation, high adsorption affinity and capacity, and specific molecular recognition, introducing the novel magnetic separation of SAs in complex food matrices. The molecular sieving effect of nanomaterials (MOFs and COFs) can help to effectively separate analytes in matrix. Targets smaller than the pore size of nanomaterials can be adsorbed, and the active units or groups in the pore can help strengthen the adsorption capacity. The pores of nanomaterials can be used to absorb SAs through weak interactions such as hydrogen bonding, van der Waals interaction, and $\pi$-$\pi$ stacking, while macromolecular substances such as proteins and polysaccharides are excluded from the pores [14, 15]. In this section, magnetic functional nanomaterials, MOFs, and COFs were highlighted for the advantages of ultrahigh sensitivity and promising adsorption affinity to present the advancement of the novel nanomaterials for the analysis of SAs.

*3.1. Extraction and Analysis by Magnetic Nanomaterials.* Numerous promising magnetic nanoadsorbent materials for the extraction of SAs in complex food matrices have been reported on the adsorption and analysis of SAs. Zinc ferrite, which has a simple and magnetic synthesis process, can efficiently and selectively extract SAs from the environment and food matrix. The method has a wide linear range (up to $250\,\mu g\,L^{-1}$), confirming the affinity of zinc for SAs, and can be used to determine SAs with concentrations higher or lower than the MRL [23]. Magnetic mesoporous polymelamine formaldehyde composites, which exhibited the combined properties of $NH_2$-$SiO_2$@$Fe_3O_4$ and magnetic properties of nanoparticles, were successfully applied to determine SAs in eggs and milk samples [24]. Magnetic dispersion SPE combined with ion pair DLLME (MDSPE-i-DLLME) was employed to detect SAs in animal food with detection limits of 0.01 to $5\,\mu g\,kg^{-1}$ and linear range of $0.1–400\,\mu g\,kg^{-1}$ [25]. SAs in water, milk, and chicken products were extracted by magnetic micro SPE (M-$\mu$-SPE) using mercaptan-functionalized magnetic carbon nanotubes and then analyzed by HPLC with diode array detector (HPLC-DAD). The linearity was good ($r^2 \geq 0.9950$) within the range of 0.1–$500\,\mu g\,L^{-1}$, and the detection limit was low ($0.02–1.5\,\mu g\,L^{-1}$) [26]. Tol-functionalized MCNT (Tol MCNT), as the adsorbent

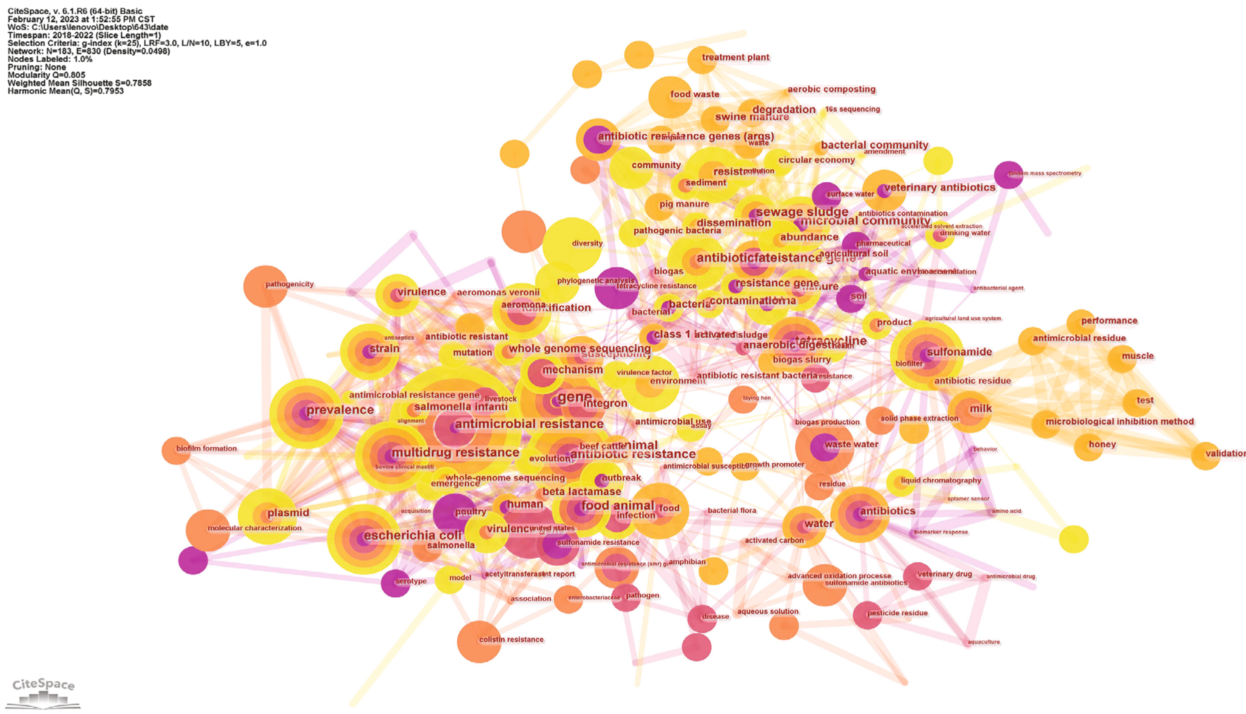


Figure 1: Keyword cooccurrence map with CiteSpace (version 6.1.R3) on November 1, 2022.

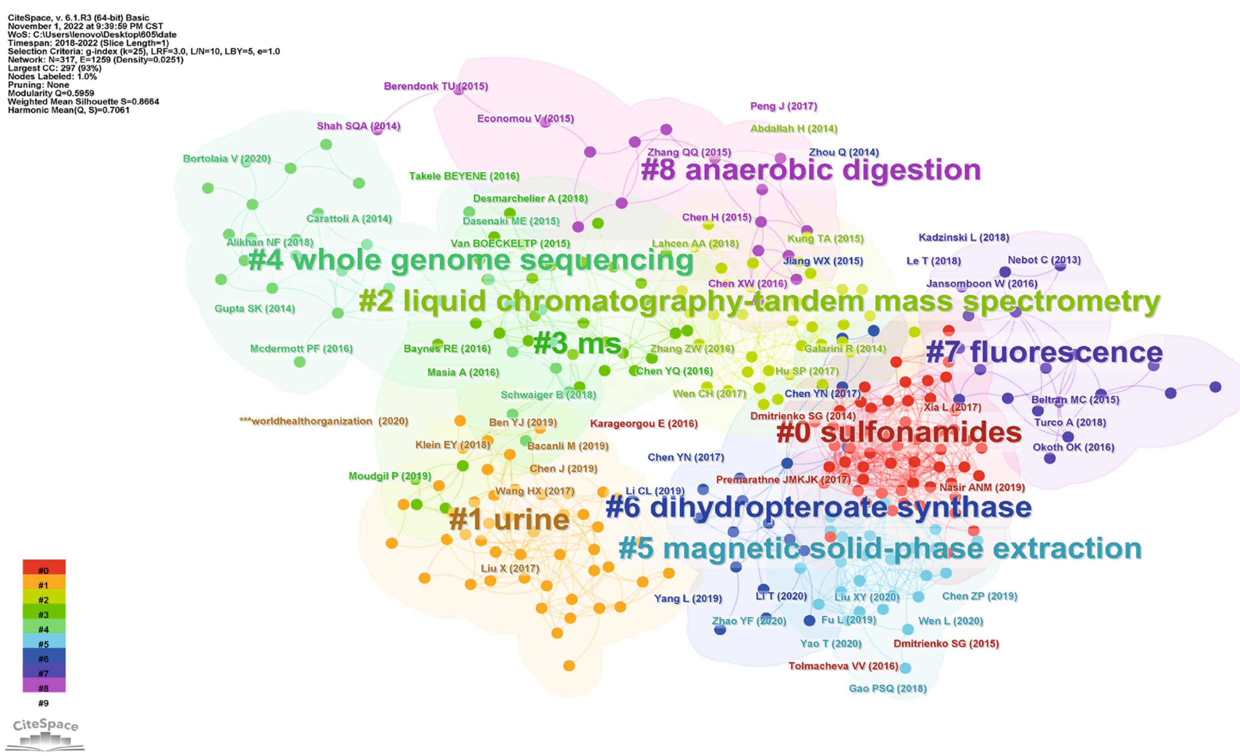


Figure 2: Cluster atlas of co-cited literatures with CiteSpace (version 6.1.R3) on November 1, 2022.

of MSPE, was combined with liquid chromatography high-resolution mass spectrometry (LC-HRMS) to analyze the concentrations of sulfadiazine in skimmed milk and whole milk samples. The detection limit of 2–10 $\mathrm{ng\,L^{-1}}$ is far lower than the MRL in milk stipulated by the European Union with the accuracy range of 92.3–101.8% [27].

The three-dimensional structure of nanomaterials can hinder their aggregation and reaggregation and leads to

Table 1: Novel materials for SA analysis with excellent performance.

| Separation materials or methods | Samples | Linear range | LOD | SAs |
|---|---|---|---|---|
| Vortex-ultrasonic-assisted dispersive liquid-liquid microextraction | Fish samples | 0.02–10 mg $kg^{-1}$ | 0.0053–0.009 mg $kg^{-1}$ | Sulfadiazine, sulfamerazine, sulfamethazine, sulfachlorpyridazine, sulfamethoxazole, sulfisoxazole, sulfadimethoxine [16] |
| Magnetic covalent organic frameworks (TpBD@$Fe_3O_4$) for microextraction | Milk and meat samples | $50 - 5 \times 10^4$ $\mu g\,L^{-1}$ | 3.39–5.77 $\mu g\,L^{-1}$ | Sulfadiazine, sulfamerazine, sulfamethazine, and sulfamethoxazole [17] |
| Solid phase microextraction with 3D graphene oxide/ lanthanum nanoparticles @ Ni foam | Animal-based food products | 0.4–700.0, 0.3–900.0, and 0.25–500 $\mu g\,L^{-1}$ | 0.14, 0.11, 0.08 $\mu g\,L^{-1}$ | Sulfadiazine, sulfamethoxazole, sulfamethazine [10] |
| Hydrophilic magnetic molecularly imprinted polymers for the dispersive solid-phase extraction | Chicken, cow milk, and goat milk | $5 \times 10^3 - 1 \times 10^4$ $\mu g\,L^{-1}$ | 0.57–1.50 $\mu g\,L^{-1}$ | Sulfadiazine, sulfathiazole, sulfamethoxydiazine, sulfamerazine, sulfamethoxypyridazine, sulfisoxazole, sulfachloropyridazine, sulfamethazine, sulfamethoxazole, sulfameter [18] |
| Monolithic covalent organic framework-based solid phase extraction | Meat products | 0.5–200 $\mu g\,kg^{-1}$ | 0.10–0.23 $\mu g\,kg^{-1}$ | Sulfamerazine, sulfamethazine, Sulfathiazole, sulfamethoxazole, n-acetyl sulfamethoxazole [19] |
| Triazine-based porous organic polymer as pipette tip solid-phase extraction adsorbent | Chicken, beef, egg, and milk | 1–300 $\mu g\,L^{-1}$ | 0.10–0.28 $\mu g\,L^{-1}$ | Sulfamethazine, sulfamonomethoxine, sulfisoxazole, sulfachloropyridazine, sulfadimethoxine [20] |
| Electrospun polyacrylonitrile/ covalent organic framework nanofibers for efficient enrichment | Spiked food samples | 0.5–50 $\mu g\,L^{-1}$ | 0.10–0.18 $\mu g\,L^{-1}$ | Sulfadiazine, sulfamethoxypyridazine, sulfisoxazole, sulfamonomethoxine, sulfamethoxazole, sulfamethazine, sulfachloropyridazine, [21] |
| Nanostructured polyaniline-based pipette tip solid-phase extraction | Milk, honey | 0.05–50 $\mu g\,L^{-1}$ | 9.2–13.5 $\mu g\,L^{-1}$ | Sulfadiazine, sulfapyridine, and sulfamethoxazole [22] |

better stability, which is conducive to the exposure of more active sites. $Fe_3O_4$@n$SiO_2$ surface coated with three-dimensional and flower-like $SnS_2$ materials $Fe_3O_4$@n$SiO_2$-$SnS_2$ synthesized by in situ growth method (Figure 3) can achieve the extraction and desorption equilibrium of target SAs within 3 minutes [28]. The MSPE method for the purification of magnetic carbon nanotube pseudomolecularly imprinted polymers was used to study the separation and enrichment of SAs in fish and shrimp samples [29]. Magnetic adsorbent $Fe_3O_4$/reduced graphene oxide carbon nanotubes have a large specific surface area and good magnetism, which are suitable for MSPE and HPLC determination of SAs [30]. After surface carboxylation of magnetic graphene oxide nanoparticles, magnetic nanoadsorbent materials were prepared. The enrichment coefficients of eight SAs after elution were 1320–1702, showing great potential for concentrating trace organic pollutants in complex matrices [31].

Core–shell magnetic nanocomposites ($Fe_3O_4$@$MoS_2$) as an adsorbent have been successfully applied to the extraction and analysis of SAs in water, milk, pork, and fish [32]. Spherical shell-type $Fe_3O_4$@graphitic carbon (YS-$Fe_3O_4$@GC) with an adjustable inner cavity was fabricated. The suitable inner cavity, graphite carbon shell, and large specific surface area of the analyte play a great role in improving its mass transfer. For the MDSPE material YS-$Fe_3O_4$@GC, the submicron box presents excellent enrichment performance for SAs and can be used for the sensitive/synchronous detection of trace SAs in milk and meat samples. This work not only provides a simple strategy for preparing tunable core–shell structures but also recommends conditions for their successful exploration as MDSPE materials [33]. An MSPE-DLLME-ultrahigh performance liquid chromatography- (UHPLC-) tandem mass spectrometry method was developed based on polypyrrole-modified magnetic multiwall carbon nanotube composites for the simultaneous quantification of eight sulfonamide antibiotics in environmental water samples. The method has good adsorption performance, with detection limits of 1.02–2.97 ng $L^{-1}$, quantification limits of 3.05–9.89 ng $L^{-1}$, and high sensitivity [34]. The Cu@PPy @HNTs@$Fe_3O_4$ composite (Figure 4) can adsorb SAs through hydrogen bonding, hydrophobic, $\pi$-$\pi$, and $\pi$-electron–metal interactions and has a response range of 2.5–100.0 $\mu g\,kg^{-1}$ to sulfamethoxil. The material has a detection limit of 2.5–5.0 $\mu g\,kg^{-1}$ as well as good reproducibility and reusability [35].

*3.2. Extraction and Analysis Based on MOFs.* MOFs are porous materials synthesized through different interactions between metal and organic connectors and possess a large surface area, high adsorption capacity, high stability, controllable structure, adjustable porosity, hierarchical structure, and recyclability [36, 37]. MOF matrix composites

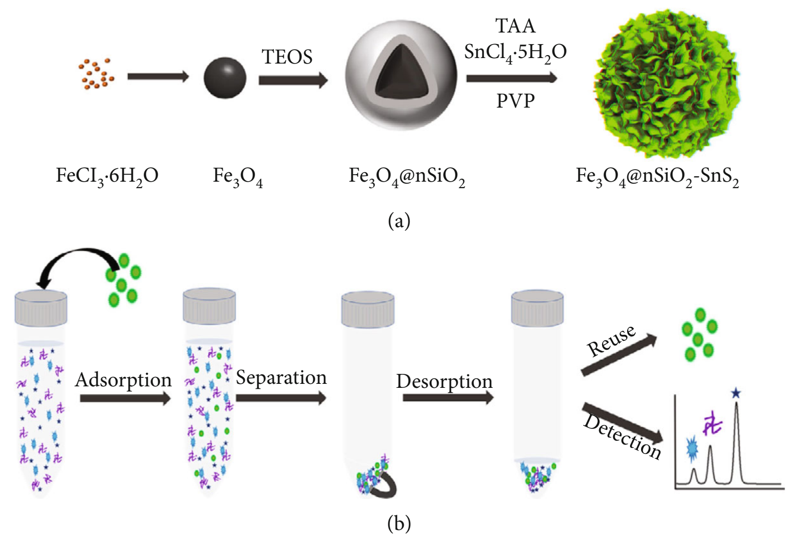


FIGURE 3: Fabrication of $Fe_3O_4$@$nSiO_2$-$SnS_2$ (a) and the analysis based on MSPE (b) [28].

exhibit acceptable performance for pollutant analysis [15, 38]. Porphyrin metal–organic skeleton (PCN-224) was used as an adsorbent in liquid chromatography-tandem mass spectrometry for the quantitative analysis of ultratrace polar SAs in food and drinking water with the advantages of a low detection limit, wide linear range, good repeatability, and reproducibility [39].

The extraction performance of mesoporous MOFs UiO-67 for SAs was significantly improved due to the $\pi$-$\pi$ interaction, hydrogen bonding, hydrophobic interaction, and size matching between the analytes and adsorbents. The detection method of SAs established by combining the material with HPLC analysis has the advantages of wide linearity (14.6–250 ng $g^{-1}$) and low detection limit (0.7–6.5 ng $g^{-1}$). In addition, UiO-67 is expected to be an excellent adsorbent for SPE of SAs from other complex matrices due to its mesoporous structure, Zr cluster center, and stability [40]. In recent years, functional MOF composites have gained increasing attention for the adsorption and analysis of complex matrices because of their characteristics of low toxicity and environmental friendliness. As a kind of green and renewable skeleton material, cyclodextrin metal–organic skeleton (CD-MOFs) prepared from $\gamma$-cyclodextrin ($\gamma$-CDs) and alkali metal cation showed high adsorption capacity and selectivity for SAs. The CD-MOF adsorbent had good enrichment ability for SAs, including sulfathiazoles and sulfamethoxylazine, and short adsorption equilibrium time (30 min) [41].

*3.3. Extraction and Analysis Based on COFs.* COFs are porous crystalline polymers that can carefully integrate organized building blocks into ordered structures with atomic precision through strong covalent bonds. COFs have a precise customization function and predesigned structure. Compared with traditional crystalline porous solids (i.e., MOFs and inorganic zeolites), COFs can achieve chemical and structural control of specific functions. In addition, COFs have the characteristics of adjustable pore size and structure, permanent porosity, high surface area, thermal stability, and low density. Given these advantages and the development of stable COFs in water media and even under harsh conditions, its application potential in environmental fields has gradually attracted attention [42–44].

TpBD@HPLC has a high extraction efficiency due to the $\pi$-$\pi$ and electrostatic interactions between the benzene ring structure of TpBD and the molecules of sulfa; the composite can be used in combination with HPLC to extract and detect SAs, including sulfadiazine, sulfadimethazine, and sulfamethoxazole, in milk and meat [17]. Other studies proved the use of core–shell magnetic COFs with HPLC microspheres as the magnetic core, TpBD COFs as the adsorption shells, and SPE adsorbents for the analysis of complex food samples. The method exhibits rapid detection performance and high sensitivity for the simultaneous detection of 10 kinds of SAs, with a detection limit between 0.28 and 1.45 mg $L^{-1}$. HPLC@TpBD core–shell adsorbent also shows good stability, robust SPE preconcentration capacity, excellent determination recovery, and good reusability [45]. Spherical triphenyldimethoxy p-phthalaldehyde COFs (TPB DMTP COFs) synthesized by a room-temperature method have good adsorption performance for trace polar SAs in food and water due to their excellent acid–base stability, large specific surface area, low skeleton density, inherent porosity, and high crystallinity. The method based on TPB DMTP COFs has a low detection limit (0.5–1.0 ng $L^{-1}$), wide linearity (5–1000 ng $L^{-1}$), and good repeatability (2.5%–8.7%). TPB-DMTP COFs have good application prospects for highly sensitive analysis of multiple pollutants, including SAs, in complex matrices [46].

Polyacrylonitrile/covalent organic skeleton TpBD nanofiber (PAN/TpBD) has good chemical stability, high flexibility, porous fiber structure, and excellent extraction efficiency. This material was used as an adsorbent for membrane microextraction (TFME) of seven SAs in animal-derived food samples. Under the optimal conditions, the detection limit of TFME-HPLC is 0.10–0.18 ng $mL^{-1}$, and the linear range is wide (0.5–50 ng $mL^{-1}$). Hence, PAN/

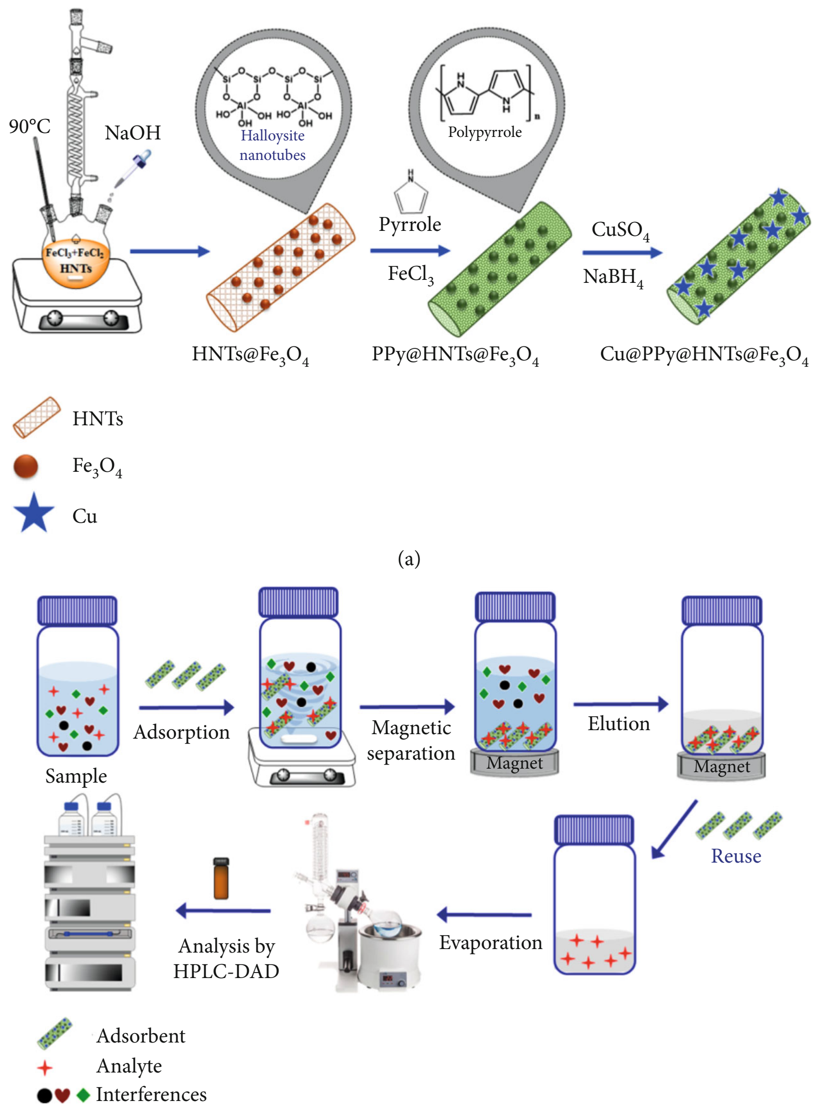


FIGURE 4: Cu@PPy @synthesis of HNTs@$Fe_3O_4$ composite (a) and its discrete magnetic solid-phase extraction and analysis for SAs (b) [35].

TpBD nanofibers have great potential in the efficient extraction of SAs from complex food samples [21].

A covalent organic skeleton Schiff base network-1 (SNW-1) was synthesized based on the reaction of p-benzaldehyde with melamine. The prepared SNW-1 was used as a pipette tip SPE adsorbent to extract SAs before HPLC analysis. A good linear relationship exists between 5 and 500 ng $mL^{-1}$, and the detection limit is lower than 0.25 ng $mL^{-1}$. SNW-1 has great potential to enrich trace SAs in complex matrices [47]. SNW-1 combined with polyacrylonitrile (SNW-1@PAN) nanofibers were used as pipette-tip SPE (PT-SPE) adsorbent for preconcentration of five SAs in meat samples. The three-dimensional network of electrospun nanofibers provides low back pressure and rapid mass transfer in PT-SPE applications, thereby preventing high back pressure and leakage problems caused by packaging SNW-1 directly into PT-SPE cylinders. The recoveries of five SAs in pork and chicken samples vary from 86% to 111% with good precision [48].

Core–shell HPLC@COF nanospheres were used to effectively extract and enrich SAs [49]. A simple and sensitive method for quantitative analysis of six SAs, including sulfadiazine, was established by combining them with HPLC. The method has good linearity and recovery, and the product can be used as a potential adsorbent for efficient extraction and analysis of trace SAs [50]. $\beta$-Cyclodextrin-functionalized magnetic covalent organic skeleton ($Fe_3O_4$@COF@Au-$\beta$-CD) was used as a magnetic solid-phase adsorbent to analyze trace SAs in meat samples by HPLC-MS/MS. Under the optimized conditions, the MSPE-HPLC method presents good linearity, with a detection limit of 0.8–1.6 $\mu$g $kg^{-1}$ [49]. COFs are modified on the surface of MOFs to generate new MOF@COF hybrid magnetic nanospheres used as magnetic adsorbents for the extraction of SAs from food and environmental samples [51]. Porous covalent organic nitrogen frameworks (PCONFs) are used as fillers in solid-phase extraction cartridges to rapidly extract eight SAs from complex samples. PCONFs combined with HPLC-MS/MS can simultaneously analyze multiple SAs with ultrahigh sensitivity, reliability, and cost efficiency [52].

## 4. Challenges and Perspectives

Research of magnetic nanomaterials, MOFs, and COFs has made great progress. Given the ultrahigh porosity and the highly selective ligand group modification of target analytes, these materials are suitable for the separation and analysis of many pollutants, including SAs. Magnetic nanomaterials are often used as adsorbents for the extraction and analysis of SAs because of their good recovery and sensitivity. However, relevant research is still on the laboratory scale. Further efforts are needed to validate their potential for practical applications. Although MOFs and COFs have been widely studied, they have limitations. Some MOFs or COFs are easy to decompose, which seriously hinders their application. In addition, the thermal stability of MOFs is closely related to organic ligands and metal ions. Developing smart MOFs or COFs that integrate sample preparation and pollutant detection may be a promising research direction. However, further efforts are needed to improve the detection sensitivity of MOFs or COFs. At present, research on the application of COF-based nanomaterials for SA detection in food samples needs to be strengthened.

The use of MOFs and COFs or modified nanocomposites with green synthesis provides new insights into the catalytic degradation of pollutants. Selecting appropriate materials or/and specific synthesis methods to prepare nanocomposites can effectively improve the catalysis and degradation of pollutants. The introduction of functional groups into MOFs or COFs can effectively increase their adsorption capacity. The integration between catalysts (or enzymes) and MOFs or COFs exhibits excellent synergistic catalytic activity in terms of colorimetric sensing or degradation of pollutants.

In conclusion, although magnetic nanomaterials, MOFs, COFs, or composites have broad application prospects in the adsorption and analysis of SAs, further efforts should be made to achieve the broad practical applications of these novel nanomaterials.

## Conflicts of Interest

The authors declare no conflict of interest.

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