



Natural-derived sorbents: Application of biochar materials as green extractive approach in food analysis

Carla Iglesias-Martín, Ana M. Ares, José Bernal, Adrián Fuente-Ballesteros^{*} 

Analytical Chemistry Group (TESEA), I. U. CINQUIMA, Faculty of Sciences, University of Valladolid, 47011, Valladolid, Spain

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ABSTRACT

The transition toward greener methodologies in analytical chemistry has intensified interest in biochar as a sustainable sorbent for food analysis. Derived from the pyrolysis of agro-industrial residues, biochar combines low-cost production with good properties such as high surface area, porosity, and surface tunability. This review provides a critical and feedstock-oriented overview of biochar applications in food sample preparation, categorizing sorbents based on their biomass origin (fruit waste, nut and seed residues, cereal by-products, ligno-cellulosic fibers, and wood waste). Each source is examined in terms of its physicochemical characteristics, extraction efficiency, and performance across different food matrices. Special emphasis is placed on sorbent modification strategies, the use of environmentally compatible desorption solvents, and the alignment of biochar use with green analytical chemistry (GAC) principles. Additionally, the review identifies key research gaps, limitations in analytical reproducibility, and challenges for regulatory acceptance. Overall, biochar emerges as a versatile and eco-efficient material with strong potential to enhance sustainability in food safety analysis.

1. Introduction

The increasing emphasis on sustainability in scientific research has propelled the integration of green chemistry principles into analytical workflows. In this context, the development of environmentally friendly materials and practices has become a key priority, especially in sample preparation—often the most resource- and reagent-intensive step in analytical procedures. Among the strategies to enhance sustainability, the use of natural sorbents derived from renewable sources has gained particular attention [1,2]. These materials, originating from agricultural and natural residues, align with several of the 12 principles of green analytical chemistry (GAC), contributing to safer, cleaner, and more resource-efficient methods [3]. To assess the greenness of analytical methods, several evaluation tools have been developed, such as the analytical GREENness metric (AGREE) [4], the analytical greenness metric for sample preparation (AGREEprep) [5,6], and the complementary green analytical procedure index (ComplexGAPI) [7]. More recently, other complementary metrics such as the blue applicability grade index (BAGI) [8], the red analytical performance index (RAPI) [9] or the violet innovation grade index (VIGI) [10] have been introduced, promoting a multidimensional view of analytical performance.

Within the spectrum of natural-derived sorbents, biochar stands out

as a versatile and promising material. Produced through the pyrolysis of biomass under oxygen-limited conditions, biochar exhibits a porous structure, customizable surface functionalities, and high thermal and chemical stability [11,12]. These characteristics make it suitable for use in sorptive techniques such as solid-phase extraction (SPE) and its miniaturized or magnetic variants, which are widely employed in food analysis due to their efficiency in preconcentration and matrix cleanup [13,14]. Beyond their analytical functionality, biochar-based sorbents exemplify the principles of circular economy by transforming low-value agricultural waste into high-value analytical materials [15]. This valorization of biomass not only reduces the environmental burden associated with waste disposal but also adds economic and social value to agro-industrial by-products. In regions with significant agricultural activity, this approach offers a dual benefit: minimizing environmental impact and creating local supply chains for sorbent production. The integration of circular economy principles into analytical chemistry fosters a more holistic vision of sustainability [16].

From an analytical chemistry perspective, one of the key challenges in food analysis lies in the complexity and heterogeneity of food matrices, which may contain fats, proteins, polysaccharides, and micronutrients that interfere with the accurate quantification of target analytes [17]. Therefore, the selection of a suitable sorbent is not only

^{*} Corresponding author.

E-mail address: adrian.fuente.ballesteros@uva.es (A. Fuente-Ballesteros).

driven by sustainability but also by its compatibility with diverse matrices and its ability to ensure selectivity, sensitivity, and reproducibility. Biochar's physicochemical diversity, tunable via precursor selection and activation techniques, offers a unique opportunity to tailor sorbents for specific analytical tasks [11].

Biochar has been extensively studied in various fields, such as in electroanalytical applications, due to its potential use in sensors and biosensors that promote sustainable technologies [18] and environmental remediation [19]. However, its use as a green sorbent in food analysis remains considerably underrepresented in the literature. This review addresses that gap by presenting, for the first time, a comprehensive and comparative analysis of biochar-based sorbents categorized according to their biomass origin. Unlike previous reviews that focus primarily on extraction techniques, our feedstock-oriented approach highlights how the physicochemical properties and analytical performance of biochar are linked to the nature of the precursor material. Additionally, we discuss biochar preparation, characterization, and desorption strategies using environmentally friendly solvents. The goal is to provide a critical and updated evaluation of biochar-based sorbents, showcasing their role in advancing sustainable practices within the field of food analysis.

2. Methodology of literature review

This review discusses the trends that have followed the study of agri-waste and natural materials with particular focus on biochar sorbents applied to food matrices. Scopus, Web of Science, Science Direct and Google Scholar were used as databases to search for references using the following keyword combinations: “agricultural waste-based” OR “biochar” OR “carbonized” AND “sorbent” OR “material” AND “food”. This review exclusively considered scientific research published in English from 2018 to 2025 to evaluate their relevance to the topic of this review. All articles were read and their pertinence to the subject was confirmed. The number of published articles focusing on food analysis using biochar-based sorbents has been the first issue considered (see Fig. 1A). Although in 2018 the number of publications was relatively low, in the following years, there was a consistent increase indicating a growing interest in this area, likely driven by the demand for greener, more efficient sample preparation methods, as well as advancement in sorbent materials and analytical instrumentation. When considering the food matrices analyzed, drinking water represent 32 % of all the articles included in this study (see Fig. 1B). Fruit and vegetables follow with 30 %, likely due to the wide range of food items encompassed by this category. Other beverages like milk, tea, and juices rank third, comprising 17 % of the studies, while other matrices show an even distribution of publications (6–8 %).

Moreover, the current trend in analytical chemistry emphasizes green principles in sample preparation, promoting the use of miniaturized techniques, that minimize solvent consumption, reduce waste, and enhance efficiency [20,21]. In this context, several biochar-based

sample preparation techniques have been employed in food analysis (see Fig. 1C). Regarding the distribution of these techniques, some researchers continue to use conventional methods such as quick, easy, cheap, effective, rugged and safe (QuEChERS) (5.6 %). The most commonly used sample preparation methods were SPE and magnetic solid-phase extraction (MSPE), each representing 19.4 % of the reported studies. Pipette tip-solid phase extraction (PT-SPE) was also widely employed, accounting for 13.9 % of cases, while molecular imprinted polymer-based SPE (MIP-SPE) and dispersive micro-solid phase extraction (D- μ -SPE) were utilized in 8.3 % and 11.1 % of studies, respectively. Additionally, in-tube solid-phase microextraction (IT-SPME) was reported in 11.1 % of applications, further demonstrating the relevance of miniaturized approaches. Less frequently employed techniques included μ -SPE at 5.6 %, thin-film microextraction (TFME) and matrix solid-phase dispersion (MSPD), both at 2.8 %. The predominance of traditional SPE methods highlights their well-established applicability; however, the growing interest in miniaturized and eco-friendly extraction techniques, such as PT-SPE, μ -SPE, and IT-SPME, underscores the ongoing shift toward sustainable analytical methodologies.

3. Natural-derived sorbents

Natural-derived sorbents represent a green alternative to conventional sorbents commonly used in extraction procedures during sample preparation. Agricultural and food industry wastes constitute a diverse range of organic byproducts generated during primary production, processing, and consumption, such as crop residues (e.g., straw, husks, stalks), fruit waste, vegetable waste, and agro-industrial by-products. It is estimated that in 2022, approximately 1050 million tons of food were wasted globally across the retail sector, food services, and households [22]. This highlights the importance of its reutilization in other fields, including analytical chemistry. The integration of biowastes and naturally derived materials into sample preparation aligns with the principles of GAC by promoting the use of sustainable sorbents and reducing environmental contamination [23]. Traditional sorbents, such as silica-based and polymeric materials, often exhibit high costs and limited reusability. In contrast, natural-derived sorbents offer a versatile and a cost-effective alternative with minimal secondary pollution, leveraging their inherent abundance of functional groups and the abundance of agricultural and food waste precursors [24]. Agricultural byproducts—such as fruit peels, straw, and bracts—exhibit unique surface properties that enhance their adsorption capabilities [25]. Research in this area prioritizes improving sorbent selectivity, extraction performance, and overall stability under thermal, chemical, and mechanical conditions. Emerging trends underscore the growing application of natural sorbents derived from agricultural waste in analytical sample preparation. One of the main applications of this natural-based sorbent is for environmental remediation and water purification due to their effective extraction capability and biodegradability [24].

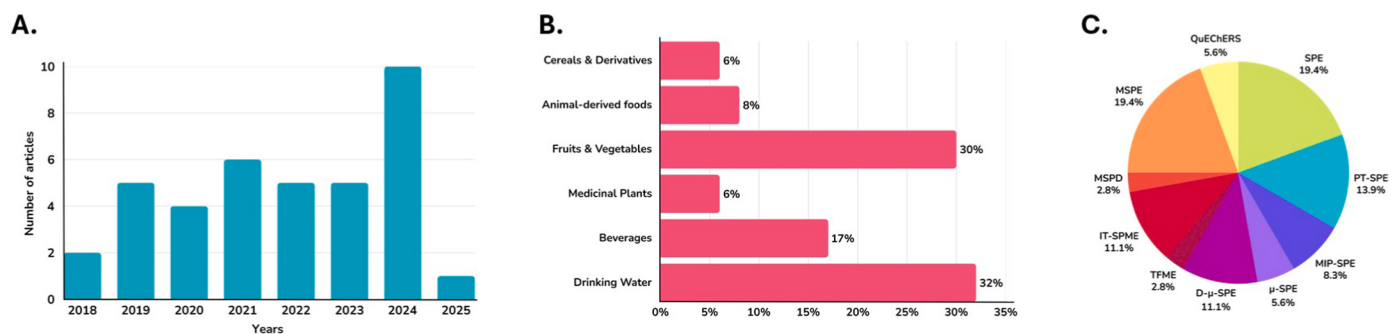


Fig. 1. A. Time trend of biochar-based sorbents applied to analysis of food matrices; **1B.** Frequency of food matrices analyzed through biochar-based sorbents; **1C.** Distribution of sample preparation biochar-based techniques with food applications.

Another field in which these green sorbents have been employed is food analysis. In this context, multiple sorbent sources, including crop waste and marine-derived materials, have been applied to the analysis of honey, tap water, juice, rice flour, and peanut oil, primarily for the study of pesticides, heavy metals, and pharmaceuticals.

Pesticides have been the target analytes in many food and drinking water analyses. A sorbent based on cork and Fe_3O_4 was employed for the extraction of several pesticides from water samples, including tap water, prior to gas chromatography coupled with electron capture detection (GC-ECD), obtaining a recovery range of 90 %–104 % [26]. The authors investigated the reusability of this magnetic sorbent and reported that it could be used three times without a substantial impact on the analytical response. They also assessed the greenness of the method, obtaining a score of 0.54 by AGREE metric [4,26]. Several studies have focused on the determination of triazole fungicides in water and honey samples. An eco-friendly sample preparation method developed by Khiaophong et al. [27] was based on the use of a modified peanut shell as an efficient sorbent for the extraction of triazole residues via μ -SPE prior to their analysis by high performance liquid chromatography (HPLC). The method achieved satisfactory recoveries ranging from 70 % to 119 %, along with low limits of detection (LOD) (0.03 $\mu\text{g/L}$ for all analytes). Additionally, Kachangoon et al. [28] utilized *Moringa oleifera* seeds to produce a selective sorbent for the enrichment of triazole fungicides in environmental water, honey, and fruit juice samples. A vortex agitator was employed to enhance the contact between the analytes and the sorbent, improving the mass transfer rate and, consequently, increasing the extraction efficiency. Under optimal conditions, this method achieved low LOD (30–50 $\mu\text{g/L}$) and acceptable relative recoveries ranging from 70 % to 112 %.

Heavy metals such as Pb^{2+} and Cd^{2+} have been analyzed in food and water samples. Ahmed et al. [29] utilized *Luffa cylindrica* sponge modified with magnetic iron oxide and TiO_2 nanoparticles as a sorbent for the MSPE of Pb^{2+} from tap water and a diverse range of food products, including baby food, cocoa powder, and black tea. The LOD for Pb^{2+} determination in liquid samples was 0.04 $\mu\text{g/L}$, while the limit of quantification (LOQ) was 0.13 $\mu\text{g/L}$. For solid samples, the LOD and LOQ were 0.159 ng/g and 0.529 ng/g, respectively. The method achieved a recovery rate of 95 % [29]. Also, for the extraction of Pb^{2+} *Spirulina maxima* has been utilized in the development of hybrid sorbents composed of graphene oxide [30]. In this study, commercial mineral water and fruit juices were analyzed using the μ -SPE technique prior to detection by ultraviolet–visible spectroscopy (UV–Vis). The method achieved a LOD of 1 $\mu\text{g/L}$ and recoveries ranging from 95 % to 101 %. Additionally, the authors assessed the environmental sustainability of the μ -SPE method using the AGREE, obtaining a score of 0.62 [30]. In a different case, for the preconcentration of trace levels of Cd^{2+} in water samples, including tap and river water, as well as rice flour samples, *Descurainia Sophia* seeds were employed as an efficient and environmentally friendly adsorbent in SPE prior to determination by flame atomic absorption spectrometry (FAAS) [31]. Khodarahmi et al. [31] reported recoveries ranging between 98 % and 106 %. Mercury (II) has also been determined in drinking water samples using an in-syringe membrane SPE procedure, employing eggshell membrane as a sorbent [32]. This method achieved good sensitivity, with a LOD of 0.44 $\mu\text{g/L}$ and a LOQ of 1.32 $\mu\text{g/L}$, as well as excellent recoveries between 89 % and 100 % when analyzed by cold vapor atomic absorption spectrometry, demonstrating the potential of animal-derived residues for heavy metal capture in aqueous media. The effectiveness of eggshell membrane as a natural sorbent for mercury is attributed to its sulfide-rich proteins, which play a crucial role in mercury adsorption [32].

Pharmaceutical compounds have also been extracted from food and water samples using natural sorbents. Han et al. [33] designed an in-syringe SPE method utilizing cotton fibers as a sorbent for the extraction of trans-resveratrol in edible oil. The methodology provided recoveries ranging from 94 % to 104 % when analyzed by HPLC–UV–Vis, demonstrating the utility of plant fibers for extracting

phenolic compounds in lipidic matrices. Rojas-Candia et al. [34] explored the rotating-disk sorptive extraction (RDSE) technique with biochar derived from laminar cork for the retention of ibuprofen and 1-hydroxyibuprofen in drinking and river water. Detection by gas chromatography–tandem mass spectrometry (GC–MS/MS) showed a recovery range of 39 %–118 % and LODs of 9–12 ng/L, indicating efficiencies comparable to commercial phases, yet highlighting the need for further optimization.

4. Biochar as green materials

An alternative use of these natural waste materials is their conversion into biochar, using them as raw materials. Biochar is a carbon-rich solid typically produced through pyrolysis of dried biomass under anoxic conditions at high temperatures. Often referred to as “black gold,” the term “biochar” originates from the combination of “bio,” derived from “biomass,” and “char,” from “charcoal” [35]. Over the past decade, the number of publications on biochar has increased significantly, highlighting a growing global interest in its potential applications. Biochar has been extensively investigated across various disciplines, with a strong focus on environmental remediation; however, food analysis remains a relatively new sector to explore.

In the field of analytical chemistry, biochar-derived sorbents have gained attention due to their efficiency in SPE techniques, low-cost production, and reduced environmental footprint [12]. Unlike traditional synthetic sorbents, often produced through environmentally unsustainable processes, biochar-based sorbents offer a green perspective as they are categorized as a renewable and biodegradable alternative, reducing their overall ecological impact. Common modification strategies—including physical, mechanochemical, and chemical treatments—improve biochar's physicochemical properties by altering its surface area, functional groups, pore structure, and size distribution [12]. The choice of modification method and treatment conditions directly influence adsorption performance and mechanisms, making biochar a versatile eco-friendly material for analytical applications such as pharma, environmental, and food analysis [11]. The extraction mechanism primarily involves a combination of physisorption and chemisorption processes. Interactions between the biochar surface and target analytes can occur through van der Waals forces, π - π stacking, hydrogen bonding, electrostatic attraction, and pore-filling effects. These interactions are governed by both the chemical nature of the analytes and the structural characteristics of the biochar, such as its aromaticity, functional group density, and porosity [36].

The reusability of biochar-based sorbents is a crucial factor in determining their sustainability and alignment with GAC principles. A higher number of reuse cycles directly contributes to reducing waste generation, minimizing the need for additional sorbent synthesis, and lowering overall resource consumption. The results of these reusability studies are summarized in Fig. 2, which visually illustrates the environmental advantages of employing biochar-based sorbents in green sample preparation methodologies. Among the reviewed materials, biochar derived from bamboo and corncob demonstrated the highest reusability, with up to 100 cycles [37], indicating their exceptional stability and long-term applicability. This remarkable durability not only enhances the cost-effectiveness of the method but also significantly reduces environmental impact by extending the lifespan of the sorbent, thereby decreasing the demand for raw materials and energy-intensive production processes. Similarly, biochar obtained from cotton fibers and glucose exhibited high reusability (90 and 80 cycles, respectively) [38, 39], further reinforcing the potential of renewable biomass sources as sustainable alternatives to conventional synthetic sorbents. Other biochar sources, such as pomelo peel and kapok fibers, demonstrated reusability of up to 30 times [40,41], while another study reported a reusability of 15 times for pomelo peel-based biochar [42]. This variability in reusability observed for pomelo peel-based biochars suggested that preparation and activation conditions are critical in determining the

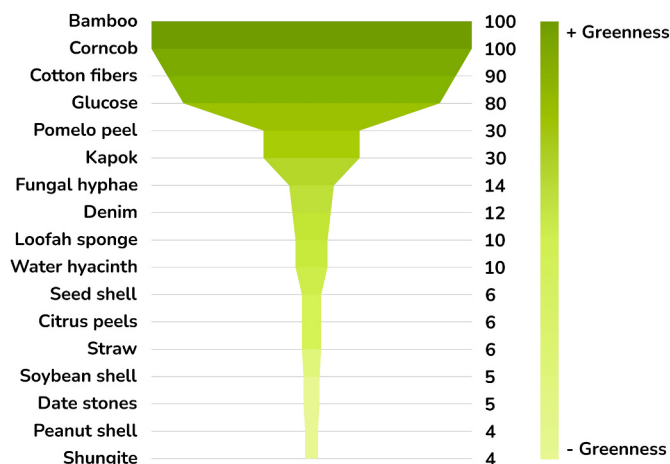


Fig. 2. Number of reuse cycles reported for biochar-based sorbents from various natural sources.

long-term stability of these materials. Fungal hyphae-based biochar (14 cycles) [43], as well as denim-derived sorbents (12 cycles) [44], also displayed moderate reusability, highlighting the viability of textile and biological waste materials in the development of eco-friendly extraction methods. Lower reusability values were reported for loofah sponge and water hyacinth biochars (10 cycles) [45], *Zizyphus jujuba* seed shells [46], citrus peels [47], and straw (6 cycles) [48], as well as soybean hulls and date stones (5 cycles) [49,50]. Biochars derived from peanut shells and shungite exhibited the least reusability (4 cycles) [36,51], indicating that certain agricultural and mineral-based sorbents may have limitations in maintaining performance over multiple uses. Despite these lower values compared to the aforementioned works, their application still aligns with GAC by utilizing renewable or naturally abundant materials rather than synthetic alternatives. These findings emphasize the critical role of sorbent selection in designing greener analytical methods. By prioritizing highly reusable biochar, the overall environmental footprint of the analytical workflow is minimized, reducing both material waste and energy consumption associated with sorbent production.

4.1. Biochar preparation

Biochar is produced through the thermal decomposition of biomass under limited or no oxygen conditions. This method prevents complete combustion, allowing the formation of a carbon-rich material with high porosity and surface functionality [35]. As can be seen in Fig. 3, the production of biochar involves several key steps, starting with the selection of biomass feedstock, which significantly influences the final material's properties. Common feedstocks include agricultural residues, lignocellulosic biomass, wood waste, and textile fibers. Prior to pyrolysis, pre-treatment such as drying, grinding, or chemical washing is often applied to standardize particle size and enhance specific characteristics. The thermal conversion process typically employs pyrolysis, which can be classified into two types: fast pyrolysis and slow pyrolysis, depending on the heating rate, particle size, retention time, and temperature [35]. Fast pyrolysis is the intense heating of biomass feedstock at 600–1000 °C in an inert atmosphere and brief residence times of

0.5–5 s. In comparison to fast pyrolysis, slow pyrolysis has a longer heating duration up to many hours or days, and a slower heating rate [35]. Slow pyrolysis is preferred for biochar production as it yields a higher proportion of biochar compared to syngas and bio-oil. Its slower biomass degradation promotes the formation of a stable matrix after decomposition, which effectively retains volatile compounds and enhances biochar stability [35,52]. Finally, biochar undergoes posttreatment and activation processes to improve its structural properties, surface characteristics, and adsorption efficiency, making it more suitable for specific applications. These modifications include physical activation through gas treatment or ball milling, as well as chemical activation using acid and alkali treatments with reagents such as H_3PO_4 and KOH to enhance porosity and surface functionality [35].

4.2. Biochar characterization techniques

Characterization techniques are an important aspect in the preparation of biochar-based sorbents to evaluate their physicochemical properties and ensure their suitability for specific analytical applications. In general, biochar materials are characterized using these techniques to provide insights into surface area, pore size and distribution, functional groups, and adsorption capacity, all of which directly influence the sorbent's adsorption capacity and selectivity. Various techniques have been employed to characterize the structural and physicochemical properties of biochar-based sorbents. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) provide insights into surface morphology and microstructural features [49,53]. Additionally, Raman spectroscopy, X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX/EDS), and X-ray photoelectron spectroscopy (XPS) are commonly used to examine the crystallinity, elemental composition, and surface chemistry of biochar materials [39,42]. For textural characterization, the Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) methods, along with nitrogen adsorption-desorption isotherms, are widely utilized to determine specific surface area and pore structure [54, 55]. Fourier-transform infrared spectroscopy (FTIR) is extensively applied for the identification of surface functional groups [46]. In addition, other techniques such as thermogravimetric analysis to assess the thermal stability of biochars [48], vibrating sample magnetometry (VSM) [53,56], dynamic light scattering (DLS) [53] and elemental analysis (EA) are frequently used to assess thermal stability, magnetic properties, particle size distribution, and the chemical composition of biochar, including total carbon, nitrogen, and hydrogen content [40].

These characterization techniques enable researchers to optimize biochar properties for enhanced performance in SPE and other separation methods. By tailoring the sorbent's structure, these analyses facilitate improved interactions with target analytes, leading to higher extraction efficiency, greater reproducibility, and increased material durability. Ultimately, comprehensive characterization is critical in developing biochar-based sorbents that are both cost-effective and environmentally sustainable. It is common practice to employ a combination of characterization techniques to comprehensively analyse biochar materials, as each method provides complementary information. As shown in Fig. 4, 87 % of the studies employed SEM to examine pore structure, solid microstructure, and morphological characteristics. Additionally, 64 % of the reviewed articles used FTIR to identify functional groups based on characteristic vibrational transitions and 44 % of



Fig. 3. Main steps in the synthesis and activation of biochar-based sorbents.

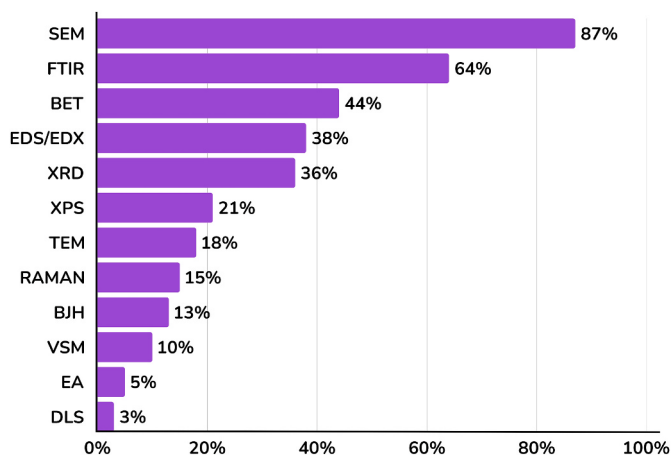


Fig. 4. Frequency of use of characterization techniques in the reviewed articles.

the articles used the BET method.

4.3. Solvents for desorption

In sample preparation, selecting appropriate solvents is vital for achieving analytical efficiency while minimizing environmental and health risks. The extensive use of solvents, particularly in large quantities, raises concerns regarding both their ecological footprint and operator safety [57]. GAC principles emphasize the importance of either eliminating solvents whenever possible or significantly reducing their use by opting for safer alternatives, such as microliter amounts of environmentally friendly solvents [23]. The assessment of solvent sustainability in the reviewed studies was conducted using the CHEM21 solvent guide [58], with the results summarized in Fig. 5. Among the solvents analyzed, the most frequently employed were acetonitrile (ACN) (36 %) and methanol (MeOH) (16 %). ACN, was often applied individually or in high-percentage mixtures [46,59]. MeOH, ranking second in usage, presents a lower environmental risk but is still commonly used in different works [36,46,60]. Water (12 %), while less frequently used as a primary solvent, was typically combined with ACN or MeOH often in high proportions [39,47,61], though some studies reported its use at concentrations of around 20 % [37,62]. Despite a noticeable shift toward greener solvent choices, the review also highlighted cases of highly hazardous solvent use. For instance, hexane, despite being classified as highly hazardous, was reported in small volumes (350 μ L) [63]. Similarly, although dichloromethane (DCM) presents significant environmental and health concerns, 16 mL were

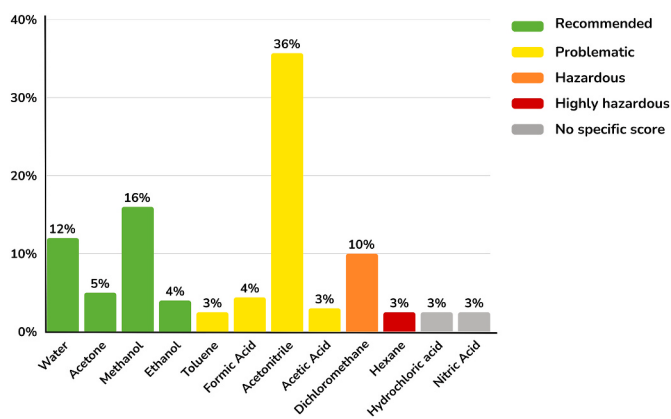


Fig. 5. Frequency of elution solvents in biochar-SPE-based sample preparation methods in food matrices and their classification category according to CHEM21 [58].

used to analyse benzo[a]pyrene derivatives from fish, followed by HPLC-UV [64].

4.4. Use of biochar as sorbent in food analysis

A wide range of biochar sources have gained attention in analytical chemistry, particularly in environmental, food, and pharmaceutical analysis [11]. Focusing on the applications of biochar-based sorbents derived from agricultural waste and related materials for the analysis of food and drinking water, multiple feedstocks have been investigated for their efficiency in sample preparation techniques. The most notable raw materials reported in the literature include: (i) fruit waste (33 %), (ii) nut residues (13 %), (iii) cereal by-products (10 %), (iv) lignocellulosic fibers (28 %), (v) wood waste (5 %), and (vi) other natural residues (10 %). Among these, sorbents derived from fruit waste have received the most attention, as illustrated in Fig. 6, likely due to their abundance, renewable nature, and favorable physicochemical properties for adsorption. These biochar-based materials have demonstrated promising performance in extracting contaminants, including pesticides, heavy metals, and pharmaceutical residues, from complex food and water matrices. The following sections discuss recent advances in the use of biochar derived from raw materials for sample preparation, highlighting their analytical performance, synthesis strategies, the use of solvents for desorption and reusability.

4.4.1. Fruit waste

Fruit waste has gained significant attention due to its versatility, as various parts such as peels, trunks, pseudo-stems, leaves, and piths can be utilized, even though these components generally lack effective post-harvest applications for farmers. The use of biochar-based sorbents derived from fruit peels and seeds has been reported for the extraction of a wide range of pesticides [42,47,55,65]. These natural materials represent a promising alternative to conventional synthetic sorbents, as they have been the most abundant biochar source over the past six years. Their application has enabled the development of analytical methods with remarkable performance characteristics, further supporting their potential in green sample preparation strategies (see Table 1).

Banana peel is particularly valuable as a carbon-rich material, containing high amounts of cellulose, hemicellulose, pectin, and chlorophyll pigments [66]. Sorbents derived from banana waste have been applied for the extraction and enrichment of pesticides from vegetable and fruit samples, achieving LOD of 0.03–10 μ g/L and recoveries in the range of 64–133 %, comparable to commercial sorbents [66]. In this study, sixteen activated biochars derived from banana peels were designed and synthesized, incorporating modifications under basic, acidic, and metallic salt conditions. The preparation process considered four key parameters: initial activation temperature, treatment reagent, impregnation ratio, and secondary activation temperature, assessing their combined effects on biochar properties. The resulting biochars were applied to pipette-tip micro-solid phase extraction (PT- μ -SPE), and their efficiency was evaluated by extracting twelve pesticides. The extracted compounds were subsequently analyzed using ultra-high-performance liquid chromatography tandem mass spectrometry (UHPLC-MS/MS) with an electrospray ionization (ESI) source. The banana peel-derived biochar that proved to be the most suitable as an extractive phase was the one prepared under acidic treatment, with an impregnation ratio of 1:1 and an initial activation temperature of 450 $^{\circ}$ C. To assess real-world applicability, five fruiting vegetables were screened for potential pesticide contamination [66].

Mangosteen peel has also been successfully utilized for the extraction of flavonoids from natural supplements using MSPD [62]. Peng et al. [62] prepared the biochar by carbonizing mangosteen peel in a muffle furnace at 450 $^{\circ}$ C for 2 h. The resulting material was then ground with solid K_2CO_3 and further activated at 900 $^{\circ}$ C. Following extraction, the target analytes were eluted with 200 μ L of solvent using an SPE vacuum manifold and subsequently analyzed by ultra-high-performance liquid

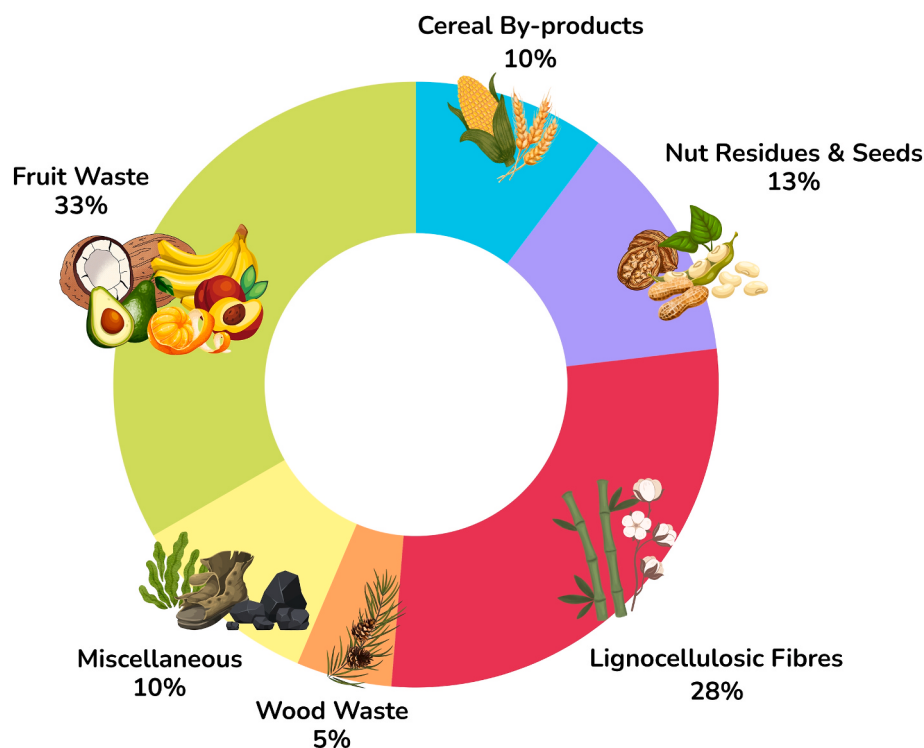


Fig. 6. Frequency of raw materials used in preparation of biochar.

Table 1

Recent studies utilizing fruit residue-derived biochar in food commodity analysis.

Sample prep.	Biochar source	Target analytes	Food matrix	Elution solvent	LOD ($\mu\text{g/L}$)	Detection	Recovery %	Ref.
D- μ -SPE	Avocado seeds	OCPs	Apple, mango, orange juice	Acetone	0.02–0.69 ^b	GC-MS	87–100	[55]
US-MSPE	Nectarine core	PAHs	Tomato paste	DCM	0.028–0.053 ^c	GC-MS	88–98	[53]
SPE	Date stones	Non-steroidal drugs	Tap water	ACN	1–2	HPLC-UV	91–99	[68]
MSPE	Date stones	Non-steroidal drugs	Tap water	ACN	0.061–0.111	LC-MS	93	[50]
PT- μ -SPE	Banana peel	Pesticides	Cucumber, tomato, zucchini, eggplant, sweet pepper	ACN	0.03–10	UHPLC-MS/MS	64–133	[66]
MSPE	Melon peel	Cu^{2+} , Cd^{2+} , Pb^{2+}	Pepper, black cabbage, eggplant, tomato	HCl	0.41–3.16	FAAS	98–101	[67]
IT-SPME	Pomelo peel	PAHs	Bottled water, soft drinks	ACN:water (70:30, v/v)	0.005–0.050	HPLC-DAD	83–121	[69]
SPE	Coconut shell	Pesticides	Tap water	DCM	0.025–0.039	HPLC-DAD	58–105	[65]
MSPD	Mangosteen peel	Flavonoids	Dendrobium huoshanense	MeOH:water (80:20, v/v)	0.00387–0.159 ^c	UHPLC-QTOF/MS	80–100	[62]
QuEChERS	Citrus peels	Pesticides	Broccoli, lettuce, tomato	ACN:water (4:6, v/v)	0.01–0.19 ^a	UHPLC-MS/MS	81–117	[47]
QuEChERS	Coconut husk	Phthalate esters	Breast milk	ACN	0.012–0.020	GC-FID	84–97	[70]
MSPE	Pomelo peel	Triazole fungicides	Fruit	ACN	0.12–0.55 ^a	GC-MS	82–110	[42]
MSPE	Pomelo peels	Fluoroquinolones	Tap water	MeOH:FA (99:1, v/v)	0.12–0.46	HPLC-DAD	84–113	[40]

^a $\mu\text{g/kg}$.

^b ng/L .

^c ng/g .

^d ng/mL .

^e $\mu\text{g/g}$. ACN, Acetonitrile; DCM, Dichloromethane; D- μ -SPE, Dispersive Micro-Solid Phase Extraction; FA, Formic Acid; FAAS, Flame Atomic Absorption Spectroscopy; GC-FID, Gas Chromatography-Flame Ionization Detector; GC-MS, Gas Chromatography-Mass Spectrometry; HCl, Hydrochloric Acid; HPLC-DAD, High-Performance Liquid Chromatography-Diode Array Detector; HPLC-UV, High-Performance Liquid Chromatography-Ultraviolet Detection; IT-SPME, In-Tube Solid Phase Microextraction; LC-MS, Liquid Chromatography-Mass Spectrometry; MeOH, Methanol; MSPD, Matrix Solid-Phase Dispersion; MSPE, Magnetic Solid Phase Extraction; OCPs, Organochlorine Pesticides; PAHs, Polycyclic Aromatic Hydrocarbons; PT- μ -SPE, Pipette Tip Micro-Solid Phase Extraction; QuEChERS, Quick, Easy, Cheap, Effective, Rugged, and Safe; SPE, Solid Phase Extraction; UHPLC-MS/MS, Ultra-High-Performance Liquid Chromatography-Tandem Mass Spectrometry; UHPLC-QTOF/MS, Ultra-High-Performance Liquid Chromatography-Quadrupole Time-of-Flight Mass Spectrometry; US-MSPE, Ultrasound-Assisted Magnetic Solid Phase Extraction.

chromatography coupled with quadrupole time-of-flight tandem mass spectrometry (UHPLC–Q–TOF/MS). This approach provided a cost-effective and environmentally friendly alternative to conventional sorbents, while achieving high sensitivity, with recoveries ranging from 80 % to 100 % and LOD between 0.00387 and 0.159 µg/g [62].

Heavy metals such as Cu²⁺, Cd²⁺, and Pb²⁺ have been effectively extracted using biochar-based materials derived from fruit waste. Ozdes et al. [67] synthesized and characterized a magnetic adsorbent composed of melon peel biochar and CoFe₂O₄ nanoparticles. The sample preparation involved MSPE, utilizing a 0.1 M HCl solution for analyte elution before FAAS analysis. This method was successfully applied for the separation, preconcentration, and simultaneous determination of these metals in pepper, black cabbage, eggplant, and tomato. The LOD were 0.41, 1.82, and 3.16 µg/L for Cu²⁺, Cd²⁺, and Pb²⁺, respectively [67].

In 2018, Huang et al. [40] developed a magnetic carbon material derived from pomelo peels using a one-pot synthesis method for the extraction of polar fluoroquinolones from real water samples, including tap. The extraction was performed using MSPE, followed by sensitive determination via HPLC with diode-array detector (DAD). In tap water, recoveries ranged from 84 % to 113 %, while the LOD for the validated method were between 0.12 and 0.46 µg/L. The authors highlighted the advantages of this approach over conventional methods, emphasizing its low cost, simplicity, satisfactory sensitivity, and environmentally friendly nature [40].

Other studies have also explored the application of biochar-based materials derived from fruit waste for the extraction of various contaminants, such as non-steroidal drugs, from water samples, including drinking water [50,68]. Additionally, PAHs have been successfully extracted from bottled water, soft drinks [69], and tomato paste [53] using these biochar-based sorbents and achieving satisfactory analytical performance in terms of sensitivity, selectivity, and recovery. Phthalate esters have also been extracted from breast milk using a modified QuEChERS sample preparation method, incorporating coconut husk biochar as a dispersive solid-phase extraction (DSPE) sorbent in the cleanup process [70].

4.4.2. Nut residues and seeds

Nut residues are agricultural residues with high lignocellulose content (e.g., walnut shell, peanut shell, and soybean shell), and they have emerged as a promising precursor for biochar production due to its high carbon content, porosity, and renewability. The conversion of these byproducts into functionalized biochar sorbents aligns with sustainable waste valorization strategies. Despite their potential, the application of nut-derived biochar in food analysis remains relatively unexplored. However, recent studies have demonstrated remarkable performance in sample preparation techniques, achieving high extraction efficiencies, low LOD, and enhanced method greenness (see Table 2).

A MIP–SPE method was developed using biochar derived from soybean shells as a sorbent for the selective extraction of carbaryl from rice and corn samples [49]. The surface of activated soybean shell biochar

was initially treated with sulfuric and nitric acids to introduce hydroxyl groups. Subsequently, a coupling agent was employed to generate double bonds carbon–carbon, followed by the use of methacrylic acid as a functional monomer. Finally, through a surface imprinting technique, the activated soybean shell biochar was polymerized with carbaryl as the template molecule. The study reported a LOD of 3.6 µg/kg and an excellent recovery range of 93–101 %. The elution step was performed using a mixture of MeOH and acetic acid (8:2, v/v), which enhanced desorption efficiency while maintaining method selectivity [49].

Sun et al. [36] successfully applied PT–SPE for the extraction of endocrine-disrupting compounds from bottled water, milk, and other beverages, utilizing biochar obtained from peanut shells. This method achieved LOD ranging from 0.25 to 2.50 µg/L and recovery values between 83 % and 117 %, demonstrating high efficiency in complex liquid matrices. Notably, MeOH was used as the elution solvent, aligning with the CHEM21 recommendations for greener analytical procedures. The adoption of PT–SPE miniaturization not only reduces solvent consumption but also enhances sustainability in sample preparation [36].

A pipette tip based µ–SPE method employing walnut-derived biochar was designed for the determination of pesticide residues in wheat flour [59]. Walnut shell was modified with a metal-organic framework (MOF) to prepare efficient biochar. The procedure consisted of mixing milled walnut shell powder with Ni–MOF and pyrolyzing the mixture at 500 °C for 1 h under N₂. After cooling, the material was treated with 0.1 M HCl to remove residual Ni–MOF and ashes, then rinsed with deionized water and dried at 110 °C for 30 min. With an UHPLC–MS/MS detection system, the method reached exceptionally low LOD, spanning from 0.01 to 12.14 µg/L, while obtaining recoveries ranging from 88 % to 131 %. ACN was selected as the elution solvent, a commonly used solvent in pesticide analysis due to its strong desorption capability [59].

Seed shells, such as those from *Ziziphus jujuba*, sunflower, and other oilseeds, represent another valuable lignocellulosic waste stream for biochar production. These agro-industrial residues offer a sustainable and cost-effective alternative to conventional sorbents, leveraging their natural porosity and functional groups for enhanced adsorption properties. Although the application of seed shell-derived biochar in food analysis is still in its early stages, recent studies have demonstrated its potential for effective sample preparation, particularly in the extraction and preconcentration of contaminants.

An article published in 2023 explored the potential of biochar derived from neem tree seed shells as a sorbent for pesticide extraction in water, including drinking water, employing TFME as the sample preparation technique [71]. TFME offers several advantages, including ease of operation, minimal sorbent requirements, low consumption of organic solvents, and rapid extraction times, making it an efficient and environmentally friendly alternative to traditional methods. In this study, the extracted pesticides were eluted using ACN and GC–ECD detection was utilized for quantification, ensuring high selectivity for the target analytes. The method demonstrated high extraction efficiency, with recoveries ranging from 81 % to 121 %, indicating the effectiveness of neem-derived biochar in pesticide retention and elution

Table 2

Applications of biochar derived from nut residues and seeds in food samples.

Sample prep.	Biochar source	Target analytes	Food matrix	Elution solvent	LOD (µg/L)	Detection	Recovery %	Ref.
MIP–SPE	Soybean shell	Carbaryl	Rice, corn	MeOH:HAc (8:2, v/v)	3.6 ^a	HPLC–UV	93–101	[49]
PT–SPE	Peanut shells	EDCs	Bottled water, milk, beverage	MeOH	0.25–2.50	HPLC–UV	83–117	[36]
µ–SPE	Walnut	Pesticides	Wheat flour	ACN	0.01–12.14	UHPLC–MS/MS	88–131	[59]
PT–µ–SPE	Seed shells	Triazine herbicides	Rice	MeOH:ACN (1:1, v/v)	1.41–3.35 ^b	HPLC–VWD	96–116	[46]
TFME	Seed shell	Pesticides	Tap water	ACN	0.003–0.48	GC–ECD	81–121	[71]

^a µg/kg.

^b ng/g. ACN, Acetonitrile; EDCs, Endocrine-Disrupting Chemicals; GC–ECD, Gas Chromatography–Electron Capture Detector; HAc, Acetic Acid; HPLC–UV, High-Performance Liquid Chromatography–Ultraviolet Detection; HPLC–VWD, High-Performance Liquid Chromatography–Variable Wavelength Detector; MeOH, Methanol; MIP–SPE, Molecularly Imprinted Polymer–Solid Phase Extraction; PT–SPE, Pipette–Tip Solid Phase Extraction; PT–µ–SPE, Pipette–Tip Micro–Solid Phase Extraction; TFME, Thin-Film Microextraction; UHPLC–MS/MS, Ultra-High-Performance Liquid Chromatography–Tandem Mass Spectrometry; µ–SPE, Micro–Solid Phase Extraction.

and excellent sensitivity, achieving LOD in the range of 0.003–0.48 µg/L, highlighting its suitability for trace-level pesticide monitoring [71]. Another type of seed-based biochar was utilized by Wang et al. [46] for the PT-SPE of triazine herbicides from rice. In this study, biochar derived from *Ziziphus jujuba* seed shells exhibited high extraction recoveries, attributed to multiple interaction mechanisms. Additionally, it demonstrated superior extraction efficiency for triazine herbicides compared to commercial adsorbents, along with enhanced reusability (at least 6 cycles) [46].

4.4.3. Cereal by-products

Cereal by-products, such as corn cob and straw, are abundant agricultural residues that have gained increasing attention as precursors for the production of biochar-based sorbents. Their high lignocellulosic content and porous structure make them particularly suitable for adsorption applications in environmental and food safety analysis. By converting these residues into functionalized biochar, researchers have developed efficient and sustainable extraction methods for a variety of contaminants, including pesticides, antibiotics, and PAHs.

Ribeiro et al. [60] developed a SPE method utilizing biochar derived from corn cob for the determination of pesticides in water. The procedure for the preparation of activated biochar consisted of drying and grinding corn cobs before subjecting them to pyrolysis at 400 °C under a nitrogen atmosphere, yielding 42 % biochar. The biochar was cleaned with DCM, ethanol, and acetone using ultrasonic agitation, then filtered and dried at 80 °C for 30 h. Activation was carried out using HCl and KOH (both at 3 mol/L). Basic activation involved stirring with KOH under vacuum, followed by drying at 100 °C and heating at 400 °C. Acid activation included washing with HCl, rinsing to neutral pH, and drying at 100 °C. The analytes are desorbed using a DCM:MeOH mixture (50:50, v/v), which provides effective elution but includes DCM, a solvent not recommended due to its toxicity and environmental concerns. The method achieves LOD values between 0.01 and 0.04 µg/L, with recoveries ranging from 70 % to 115 %, highlighting its broad applicability for different pesticide compounds [60].

He et al. [72] report a novel molecularly imprinted polymer (MIP) approach employing straw-derived biochar as a stabilizer for oil-in-water (o/w) pickering emulsions. The resulting emulsion is subsequently utilized to fabricate molecularly imprinted polymer microspheres (MIPMs) for the selective extraction of tetracyclines, achieving well-controlled particle sizes and high uniformity. The MIPMs have been applied as sorbents in SPE for the isolation of tetracycline from drinking water, fish, and chicken samples. The target compound is eluted using an acidified ACN solution, which enhances desorption while ensuring compatibility with HPLC-DAD detection. The method demonstrates LOD ranging from 3.51 to 3.64 µg/kg, with recoveries between 73 % and 95 %, highlighting its potential for monitoring antibiotic contamination in environmental and food matrices [72].

Ji et al. [37] developed a corn cob-derived biochar coating for the determination of PAHs in bottled water using IT-SPME. The preparation of the corn cob-derived biochar, which plays a crucial role, was carried out drying corn cob powder at 110 °C for 12 h before carbonization at temperatures ranging from 200 °C to 500 °C for different time intervals. After cooling to room temperature, the biochar was ground, sieved to 200 mesh, and cleaned with ethanol and MeOH under ultrasonication. Finally, it was dried at 60 °C for further use. Based on the observed hardening, shrinkage, and color changes, the optimal carbonization temperature was determined to be between 300 °C and 400 °C to balance graphitization and yield. The online and automatized extraction process employs an ACN:water mixture (70:30, v/v) for efficient analyte desorption, followed by detection via HPLC-DAD. The method demonstrates LOD values between 0.003 and 0.030 µg/L and recoveries ranging from 82 % to 117 %, indicating its high sensitivity and reproducibility. Remarkably, this biochar sorbent was reused for 100 extraction cycles without a significant decline in efficiency [37].

Chen et al. [48] developed a biochar-functionalized molecularly

imprinted polymer for dispersive solid-phase extraction (MIPs-DSPE) using straw-derived biochar for the selective extraction of chlorpyrifos from tap water and several aqueous environmental samples. The straw was washed, dried, and ground before being sieved to 60 mesh. Pyrolysis was conducted at 350 °C for 35 min under a nitrogen atmosphere, with a heating rate of 10 °C/min, followed by a 6-h holding period. The resulting biochar was washed with deionized water, dried at 80 °C under vacuum, and stored in the dark. For modification, the biochar was sonicated for 5 h in a ZnCl₂/H₂SO₄/HNO₃ solution, then diluted, left to react for 8 h, filtered, and dried at 50 °C under vacuum. The MIP was synthesized using this straw-derived biochar and a non-covalent imprinting method. Elution was performed with an acetic acid:MeOH mixture (20:80, v/v), ensuring efficient desorption of the pesticide prior to HPLC with photodiode array detection (PDA) analysis. The method achieved a LOD of 1 µg/L, with recoveries ranging from 84 % to 86 %, proving reliable performance for the detection of this organophosphate insecticide [48].

4.4.4. Lignocellulosic fibers

Various lignocellulosic materials, including sugarcane bagasse, cotton, sisal, palm fiber, coconut husk, corn cob, jute, and rice husk, have been explored as sorbents for SPE. Their widespread use is attributed to the presence of functional groups inherent in their lignin and cellulose-rich structures, which enhance their adsorption capabilities. These natural sorbents have been applied in analytical methodologies for diverse purposes, such as analyte preconcentration and speciation (see Table 3). Given their renewable nature and abundance, lignocellulosic materials represent a cost-effective alternative, often sourced as byproducts from agroforestry and urban waste streams [73].

In 2024, Liu et al. [41] introduced a pipette-tip solid phase micro-extraction (PT-SPME) approach utilizing biochar derived from kapok fiber for the extraction of organochlorine pesticides (OCPs) from fruit juices, herbal extract granules, and oral solutions, followed by analysis via GC-ECD. The biochar sorbent was synthesized through pyrolysis of kapok fiber at 600 °C for 2 h under an oxygen-limited atmosphere and subsequently adhered to the inner surface of a pipette tip. Characterization revealed that the kapok fiber biochar featured a highly porous, irregular structure with pore sizes between 3 and 10 nm, enhancing its capacity to adsorb small organic compounds. The study demonstrated that kapok fiber biochar retained its structural integrity for up to 30 aspiration cycles when desorbed with ACN, MeOH, ethyl acetate, acetone, or 2-propanol. However, exposure to hexane resulted in the detachment of the biochar layer after just a few cycles. The sample preparation process was highly efficient, requiring only 150 µL of ACN and a total extraction time of approximately 3 min. The method achieved excellent analytical performance, with LOD ranging from 0.03 to 0.30 µg/L and recoveries between 88 % and 117 %, underscoring its sensitivity, simplicity, cost-effectiveness, and environmentally friendly attributes [41].

Yu et al. [61] developed a composite monolithic adsorbent using bamboo-derived biochar. This sorbent was packed into a tube and employed as an IT-SPME column for the automated extraction and determination of two coumarins in traditional medicine formulations. The extraction step was integrated online with a C18 analytical column in an HPLC system prior to UV detection. The composite monolithic adsorbent was synthesized within a stainless-steel tube via a polymerization process involving a crosslinker, porogens, and an initiator, with bamboo biochar incorporated as a functional component. The mixture was sonicated, sealed within the tube, and subjected to controlled heating to induce polymerization. The resulting adsorbent column was subsequently washed with an organic solvent using an HPLC pump to remove residual porogens. The IT-SPME method utilized an ACN:water mixture (49:51, v/v) as the elution solvent, facilitating efficient desorption of the target compounds. Reported recovery rates ranged from 99 % to 105 %, demonstrating high extraction efficiency for coumarins in complex matrices. Although the study did not specify a LOD,

Table 3

Recent studies utilizing lignocellulosic-derived biochar in food commodity analysis.

Sample prep.	Biochar source	Target analytes	Food matrix	Elution solvent	LOD ($\mu\text{g/L}$)	Detection	Recovery %	Ref.
PT-SPME	Kapok fiber	OCPs	Beverages, herbal extracts	ACN	0.03–0.30 ^a	GC-ECD	88–117	[41]
IT-SPME	Bamboo	Coumarins	Traditional medicines	ACN:water (49:51, v/v)	–	HPLC-UV	99–105	[61]
IT-SPME	Silk fibers	PAHs	Tap water, bottled water	ACN:water (70:30, v/v)	0.005–0.050	HPLC-DAD	84–114	[74]
IT-SPME	Cotton fibers	PAHs	Tap water	–	0.005–0.020	HPLC-DAD	82–119	[38]
D- μ -SPE	Cotton fibers	PAHs	Tea infusions	Toluene	0.012–0.014	GC-MS	89–110	[75]
DSPE	Cotton fibers	Isoflavones	Tap water	EtOH	0.017–0.025	LC-MS/MS	80–95	[77]
MSPE	Cotton fibers	Cd ²⁺	Water, tomato, cabbage	HNO ₃	0.21	FAAS	86–102	[76]
SPE	Loofah sponge	PAHs	Fish	DCM	2.0 ^b	HPLC-UV	95–103	[64]
μ -SPE	Water hyacinth	Insecticides	Tea	Acetone	0.02–0.1	HPLC-UV	80–107	[45]
SPE	Water hyacinth	Pesticides	Beer	MeOH:DCM (60:40, v/v)	0.05–0.08	UHPLC-MS	61–102	[78]

^a ng/mL.

^b ng/g. ACN, Acetonitrile; DCM, Dichloromethane; D- μ -SPE, Dispersive Micro-Solid Phase Extraction; EtOH, Ethanol; FAAS, Flame Atomic Absorption Spectrometry; GC-ECD, Gas Chromatography–Electron Capture Detector; GC-MS, Gas Chromatography–Mass Spectrometry; HNO₃, Nitric Acid; HPLC-DAD, High-Performance Liquid Chromatography–Diode Array Detector; HPLC-UV, High-Performance Liquid Chromatography–Ultraviolet Detection; in-syringe DSPE, In-Syringe Dispersive Solid Phase Extraction; IT-SPME, In-Tube Solid Phase Microextraction; LC-MS/MS, Liquid Chromatography–Tandem Mass Spectrometry; MeOH, Methanol; MSPE, Magnetic Solid Phase Extraction; OCPs, Organochlorine Pesticides; PAHs, Polycyclic Aromatic Hydrocarbons; PT-SPME, Pipette-Tip Solid Phase Microextraction; SPE, Solid Phase Extraction; UHPLC-MS, Ultra-High-Performance Liquid Chromatography–Mass Spectrometry; μ -SPE, Micro-Solid Phase Extraction.

the performance of this biochar-based sorbent suggests strong adsorption capabilities. Moreover, the biochar exhibited remarkable reusability, maintaining good performance for at least 100 extraction cycles [61].

Another IT-SPME approach has been proposed by Ji et al. [74] using biochar derived from silk fibers for the extraction of PAHs from tap and bottled water. To synthesize the biochar, silk fibers were cleaned, dried, and carbonized at 350 °C for 30 min. The resulting material was washed with organic solvents to remove impurities and subsequently dried. The carbonized silk fibers were then packed into a polyetheretherketone tube to construct an IT-SPME device. Various tube lengths were evaluated to optimize extraction efficiency and ensure complete desorption, with 30 cm being identified as the optimal length for further analysis. This study employed an ACN:water mixture (70:30, v/v) as the elution solvent, achieving LOD between 0.005 and 0.050 $\mu\text{g/L}$. Recoveries ranged from 84 % to 114 %, indicating good reproducibility and efficiency across different matrices. Notably, the preparation conditions of silk fiber biochar played a crucial role in adsorption and desorption performance [74]. Other studies have explored biochar-based sorbents derived from lignocellulosic residues for the extraction of PAHs from various matrices. PAHs have also been analyzed in tap water [38], tea infusions [75], and fish samples [64], underscoring the versatility of biochar-based extraction techniques across environmental and food analysis applications.

In the context of MSPE, biochar derived from cotton fibers has been employed for the sequestration of Cd²⁺ ions from water, tomato, and cabbage samples [76]. The preparation of carbonized cotton fabric involved washing, drying, and chemical activation with phosphoric acid, followed by carbonization at 500 °C under a nitrogen atmosphere. The material was then cooled, rinsed, and vacuum-dried. To obtain a composite material, the carbonized cotton fabric was combined with a metal-organic framework through an immersion and drying process. Further modification included the incorporation of magnetic nanoparticles via a co-precipitation method, followed by the polymerization of thionine to enhance functional properties. For sample treatment, 20 mg of the prepared magnetic sorbent was washed and mixed with 30 mL of sample at pH 7. The mixture was sonicated for 5 min to facilitate adsorption. The sorbent was then separated using an external magnetic field, and the liquid phase was discarded. Elution was performed using a EtOH:HNO₃ mixture (50:50, v/v) under ultrasound for 2 min. Finally, the sorbent was magnetically separated again, and the desorbed analyte was quantified using FAAS. This approach achieves a LOD of 0.21 $\mu\text{g/L}$, with recoveries ranging from 86 % to 102 %, demonstrating the suitability of this biochar-based sorbent for metal ion remediation [76]. Cotton fibers have also been employed by Benedé et al. [77] for the

preconcentration of isoflavones in tap water using in-syringe DSPE, followed by determination via liquid chromatography–tandem mass spectrometry. EtOH was used as the elution solvent, achieving low LOD (0.017–0.025 $\mu\text{g/L}$) and good recoveries (80–95 %) [77].

A biochar-based sorbent derived from water hyacinth has been utilized in SPE for the determination and quantification of 18 pesticides in lager beer [78]. Water hyacinth was dried at 50 °C for 48 h and pyrolyzed in a rotary tube furnace at 400 °C for 2 h. The resulting biochar was treated with an acid solution to remove impurities, washed with deionized water until a neutral pH was achieved, and subsequently used as an adsorbent in an SPE cartridge. The SPE cartridge was assembled using a polypropylene body, a bottom frit, 150 mg of biochar, and an upper stopcock frit to ensure a compact structure. During the SPE procedure, analytes were desorbed using a MeOH:DCM mixture (60:40, v/v), which improved recovery. The analysis was carried out using a UHPLC methodology coupled with quadrupole mass spectrometer equipped with an ESI probe. The method achieved LOD between 0.05 and 0.08 $\mu\text{g/L}$, with recoveries ranging from 61 % to 102 %, indicating variable extraction efficiency depending on the specific pesticide analyzed [78]. Another group also employed water hyacinth for the extraction of benzoylurea insecticides in tea products [45]. In this case, μ -SPE was used in combination with HPLC-UV detection, achieving a LOD of 0.02–0.1 $\mu\text{g/L}$ and recoveries in the range of 80–107 %.

4.4.5. Wood waste

Biochar-based materials derived from woody parts have been explored as sustainable sorbents due to their high carbon content (>80 %, w/w), structural stability, and well-developed porosity [79]. These materials, obtained from sources such as branches, bark, and sawdust, offer enhanced adsorption properties, making them suitable for analytical applications. Their lignocellulosic composition provides a rich surface chemistry that can be tailored for selective analyte extraction, further expanding their potential in environmental and food analysis. However, their application in food analysis remains limited, with only a few studies reported in the literature.

In 2022, a study employing MSPE was conducted for the determination of sulfadiazine in water samples prior to HPLC analysis [56]. Biochar was obtained by washing pine leaves with deionized water, drying them at 60 °C, and grinding them into small particles. The material was then pyrolyzed at 1000 °C for 2 h. The magnetic sorbent was synthesized through the chemical co-precipitation of Fe₃O₄ magnetic nanocomposites modified with this biochar pine leaves. The magnetic powder was washed, mixed with a sulfadiazine solution, and stirred to facilitate adsorption. The sorbent was then magnetically separated, and the aqueous phase was discarded. Elution was performed using MeOH

containing 5 % NH₃ under vortex agitation, after which the eluate was collected and analyzed by HPLC. This hybrid material combined the high adsorption capacity of biochar with the advantages of magnetic separation, enabling efficient analyte extraction and rapid phase separation. The developed MSPE–HPLC method exhibited LOD and LOQ of 10.7 µg/L and 35.5 µg/L, respectively, demonstrating its suitability for trace-level analysis. The method was successfully applied to the determination of sulfadiazine in spiked water samples, including drinking water, highlighting its potential for environmental monitoring and ensuring water quality compliance [56].

Assefa et al. [63] developed in 2024 a vortex-assisted D–µ–SPE technique for the extraction and preconcentration of organochlorine pesticide residues in juice samples, followed by their determination using gas chromatography–mass spectrometry (GC–MS). The method employed a novel sorbent composed of a silica-supported Fe₂O₃–modified khat leftover biochar nanocomposite, which provided an efficient and cost-effective alternative to conventional adsorbents. Khat leftovers were cleaned, dried, and ground into fine particles. The biomass underwent pyrolysis in a muffle furnace, followed by washing and drying to obtain biochar. To synthesize a biochar–metal oxide nanocomposite, the biomass was treated with an iron precursor solution under stirring. The mixture was dried, pyrolyzed, and washed, then ground and sieved to obtain the final ferric biochar nanocomposite. To enhance the dispersion of the sorbent in the sample solution, a vortex mixer was used, facilitating improved analyte–sorbent interactions. The supernatant was discarded, and the adsorbent was washed. The analytes were desorbed with n-hexane, vortexed, and centrifuged. The supernatant was collected and injected into the GC–MS system for analysis. The method demonstrated excellent sensitivity, with LOD ranging from 0.001 to 0.006 ng/mL. Furthermore, matrix-matched extraction recoveries ranged from 83.4 % to 108.3 %, highlighting the effectiveness of the sorbent in real sample analysis [63].

4.4.6. Miscellaneous

Other natural sources of biochar have been reported in the literature, which do not fall within the main groups previously discussed, such as fruit waste, nut residues and seeds, cereal by-products, or lignocellulosic fibers. Despite their heterogeneous origin, these materials share the common feature of being abundant, low-cost, and biodegradable, aligning with the core principles of GAC. Recent studies have demonstrated that a wide range of unconventional natural materials such as textile fibers, algal biomass and compostable organic waste, can be effectively transformed into biochar and applied as sorbents in SPE procedures (see Table 4).

One of these sources of biochar is textile fibers from discarded and unused clothing, aligning with the principles of circular economy. Given current trends, this represents an abundant supply of cotton fibers that can undergo pyrolysis and be converted into biochar for use as sorbents. Bakhshizadeh et al. [44] employed a green sample preparation approach

using fabric phase sorptive extraction (FPSE) combined with dispersive liquid–liquid extraction (DLLE), followed by gas chromatography–flame ionization detection (GC–FID). In this study, they used carbonized cotton textile from denim as a sorbent for the extraction of pesticide residues from fruit juices. The flexible fabric-based sorbent demonstrated excellent performance, achieving recoveries between 91 % and 103 %, while serving as a green alternative due to its non-toxic nature, being derived from natural cotton fibers and avoiding the introduction of hazardous chemicals into the extraction process. Additionally, the sorbent was reusable for up to 12 extraction cycles without significant loss of efficiency, highlighting its sustainability and cost-effectiveness. Despite these advantages, the denim-based sorbent exhibited lower specificity compared to more selective materials, such as MIPs [44]. The method was applied to various fruit juices (pomegranate, grape, and orange) and fresh produce samples (orange, kiwi, and cucumber), but no pesticide residues were detected. The proposed method was classified as environmentally friendly according to ComplexGAPI, as the fabrication of the carbonized denim sorbent required simple procedures, minimal laboratory equipment, and no toxic reagents. This study highlights the potential of upcycled textile-based biochar as a sustainable sorbent, contributing to waste valorization and GAC [44].

Wang et al. [43] developed another approach for the extraction of OCPs based on MSPE method, utilizing biochar derived from fungal hyphae. This work was applied in water, tea beverages, and traditional medicines samples, using ACN as solvent for desorption. The method achieved LOD values between 0.1 and 21.4 ng/L, with recoveries ranging from 80 % to 117 %, demonstrating its suitability for trace-level pesticide analysis [43].

In another study, Sun et al. reported the use of glucose-derived biochar in an IT–SPME method for the determination of estrogens in tap water, rainwater, and river water [39]. The sorbent consisted of TiO₂ nanorod arrays on carbon fibers functionalized with glucose-derived biochar nanospheres to enhance the extraction efficiency of the carbon fibers. Additionally, covalent organic framework nanospheres were separately introduced to functionalize the TiO₂ nanorod arrays to compare their extraction efficiency with that of biochar-functionalized nanospheres. The method employed a miniaturized and automated system with online coupling to HPLC–DAD detection. The TiO₂ nanorod arrays functionalized with biochar nanospheres exhibited superior durability and higher extraction efficiency compared to TiO₂ nanorod arrays on carbon fibers. Notably, the extraction efficiency remained unchanged after 80 extraction cycles, highlighting its robustness and sustainability. The method exhibited LOD values as low as 0.001–0.005 µg/L and recoveries between 86 % and 108 % for estrogens [39].

Zhao et al. [54] developed a SPE method exploring two biochars derived from *Chlorella* and bamboo for the extraction of tetracyclines (veterinary drugs) from animal-derived food samples. Freeze-dried *Chlorella pyrenoidosa* powder was pyrolyzed under oxygen-deficient conditions, ground, and sieved to obtain biochar granules. Commercial

Table 4
Representative works using diverse biochar sources in food commodity analysis.

Sample prep.	Biochar source	Target analytes	Food matrix	Elution solvent	LOD (µg/L)	Detection	Recovery %	Ref.
MSPE	Fungal hyphae	OCPs	Water, tea beverages, traditional medicines	ACN	0.1–21.4 ^a	GC–MS/MS	80–117	[43]
IT–SPME	Glucose	Estrogens	Tap water	ACN:water (50:50, v/v)	0.001–0.005	HPLC–DAD	86–108	[39]
SPE	Algae	Tetracyclines	Animal-derived food	MeOH:FA (9:1, v/v)	0.42–0.97	HPLC–UV	81–107	[54]
SPE	Shungite	Ponceau 4R	Tap water, wine, juice	–	1.5 ^b	HPLC–UV	94–99	[51]
FPSE–DLLME	Denim	Pesticides	Fruit juices	ACN	0.31–0.51	GC–FID	91–103	[44]

^a ng/L.

^b ml/L. ACN, Acetonitrile; FA, Formic Acid; **FPSE–DLLME**, Fabric Phase Sorptive Extraction–Dispersive Liquid–Liquid Microextraction; **GC–FID**, Gas Chromatography–Flame Ionization Detector; **GC–MS/MS**, Gas Chromatography–Tandem Mass Spectrometry; **HPLC–DAD**, High-Performance Liquid Chromatography–Diode Array Detector; **HPLC–UV**, High-Performance Liquid Chromatography–Ultraviolet Detection; **IT–SPME**, In-Tube Solid Phase Microextraction; **MeOH**, Methanol; **MSPE**, Magnetic Solid Phase Extraction; **OCPs**, Organochlorine Pesticides; **SPE**, Solid Phase Extraction.

bamboo biochar was purified through acid treatment and sequential washing until neutrality was achieved. *Chlorella* biochar, bamboo biochar, and the mixed biochar at various mass ratios were individually packed into distinct SPE column types. The characterization of the two materials showed that bamboo biochar had a higher specific surface area and average pore volume than *Chlorella* biochar [54]. HPLC–UV analysis demonstrated that, despite the excellent adsorption capacity of bamboo biochar, its elution efficiency was relatively low. However, within the hybrid biochar–based SPE model, *Chlorella* biochar enhanced adsorption selectivity and desorption efficiency. Additionally, the mixed biochar exhibited a preferential adsorption capacity for tetracycline compared to other antibiotics. The method provided LOD values ranging from 0.42 to 0.97 µg/L, with recoveries between 81 % and 107 %, ensuring reliable quantification of tetracyclines in complex food matrices [54].

Another traditional SPE approach was proposed by Alham et al. [51], employing carbonized Shungite for the determination of Ponceau 4R (synthetic colorant) in tap water, wine, and juice. Shungite is a natural material composed of a mixture of various carbon allotropes, widely used as an inexpensive and effective sorbent in scientific and food research. The sorbent based on Shungite for SPE is obtained through carbonization and subsequent activation to enhance the content of micro- and mesopores, evaluating two particle sizes. The sorbent with the smallest particle size (0.3 mm) demonstrated the highest adsorption efficiency for this synthetic dye, with HPLC–UV detection ensuring reliable quantification. The method achieved a LOD of 1.5 mg/L, with recoveries ranging from 94 % to 99 %, highlighting its effectiveness for monitoring food and beverage contamination [51].

4.4.7. Comparative assessment and overview of biochar sorbents in food applications

A comparative assessment of biochar sorbents categorized by their biomass origin was carried out to provide an overview of their functional attributes (see Table 5). Among the categories, biochars derived from fruit waste represent the most comprehensively investigated group. These materials show exceptional versatility in extracting a wide range of analytes from various food matrices, including fresh produce, fruit juices, and complex biological samples such as breast milk. They typically offer high extraction efficiencies and compatibility with miniaturized formats. Nevertheless, the extraction performance remains highly dependent on the precursor's origin and pyrolysis parameters, such as temperature, particle size, and activation technique. Mechanistic interactions between fruit-based biochars and target analytes remain

insufficiently characterized, limiting the development of predictive models for extraction performance. Biochars obtained from nuts and seeds demonstrate high selectivity, especially when enhanced with functionalization techniques such as MIPs. These advanced materials are particularly effective for multi-residue determinations, including pesticides and heavy metals. However, their applicability remains narrow, with limited validation across complex matrices. Their reusability is also limited due to irreversible adsorption or inefficient regeneration protocols, which restrict their practical value in repeated analyses. Cereal-based biochars, notably those sourced from corn cob and straw, have gained attention for their capacity to recover antibiotics and PAHs from aqueous media. Their structural integrity across multiple extraction cycles suggests a promising profile for method reproducibility. However, their application is generally restricted to water-based matrices, reducing their relevance for lipid-rich or heterogeneous food samples. Despite originating from renewable resources, the extraction workflows involving cereal biochars often require hazardous solvents, compromising their sustainability profile. Lignocellulosic fiber-derived biochars have shown strong analytical performance, especially in achieving low detection limits and high repeatability. These materials are often well-suited for automation and high-throughput screening owing to their structural homogeneity. Nonetheless, they are prone to batch-to-batch variability in surface area and porosity due to inconsistencies in raw material and processing conditions. In certain cases, activation involves corrosive chemical treatments, raising concerns about the environmental impact and practical scalability. Wood residue-based biochars, though less frequently studied in food analysis, display promising robustness under thermal and chemical stress. They are often utilized in hybrid material development, such as in composite formulations to enhance sorptive capabilities. Yet, their analytical efficiency is strongly influenced by pyrolysis conditions and elution solvent properties. Many studies fail to include comprehensive sustainability metrics, and the adoption of standardized green analytical criteria is urgently needed.

Despite these advancements, multiple knowledge gaps in this area remain. Mechanistic understanding of sorbate-sorbent interactions across food matrices is still rudimentary. Literature lacks quantitative models or theoretical frameworks linking the structural characteristics of biochar to extraction performance. Furthermore, benchmarking under standardized analytical conditions is rarely conducted, making it difficult to compare materials or replicate studies. High-complexity matrices like dairy products, emulsions, and fat-rich foods are under-explored, despite being crucial in food safety analysis. The long-term

Table 5
Comparative assessment of biochar-based sorbents by biomass origin in food analysis.

Biochar source	Biomass examples	Matrix applicability	Recovery %	Advantages	Disadvantages	Overview
Fruit waste	Banana peel, mangosteen peel, melon peel, pomelo peel, coconut husk	Fruit, vegetables, water, juice, tomato paste, breast milk	64–133	Abundant raw material; high extraction efficiency; versatile across matrices; good miniaturization compatibility	Performance highly variable depending on fruit type and preparation; limited mechanistic studies	Most studied group, good performance across matrices, highest reuse for kapok fiber
Nut residues and seeds	Peanut shell, walnut shell, soybean shell, Ziziphus jujuba seed	Rice, corn, milk, wheat flour, drinking water, juice	83–131	High selectivity (e.g., MIPs); valorization of hard-shell agro-waste; promising results in multi-residue analysis	Generally low reusability; underexplored in complex food matrices	Underexplored in food analysis, good selectivity using MIPs
Cereal by-products	Corn cob, straw	Bottled water, animal products, rice flour	70–117	Excellent reusability; effective for antibiotics and PAHs; innovative material modifications (e.g., MIPMs)	Use of toxic solvents in some protocols; few applications beyond water-based matrices	Emerging strategies like MIPMs, promising for complex matrices
Lignocellulosic fibers	Kapok fiber, bamboo, cotton, silk, water hyacinth	Fruit juice, tap water, herbal extracts, cabbage, tea	80–117	Diverse applications; good analytical performance; easy integration in automation; broad biomass sources	Limited availability of some fibers; variability in pore structure; some high activation cost	Versatile sorbents, many studies using automation and green strategies
Wood waste	Pine leaves, khat waste	Drinking water, juice	83–108	High carbon content; thermally stable; potential for hybrid material development; magnetic separability	Few studies available; solvent choice affects greenness; high pyrolysis temp may limit scalability	Least explored in food matrices, promising results with hybrid systems

MIPMs, Molecularly Imprinted Polymer Microspheres; MIPs, Molecularly Imprinted Polymers; PAHs, Polycyclic Aromatic Hydrocarbons.

functional stability of biochar such as resilience to humidity, variable pH, or aging has yet to be adequately addressed. These factors directly affect their shelf life, transportability, and practical deployment. Moreover, biochar's multifunctional potential remains untapped. These auxiliary properties could offer added analytical value, provided they are subjected to robust validation and tailored for specific applications. Technically, the lack of reproducibility in biochar production is a major limitation. Parameters such as ash content, pore structure, and elemental composition are highly variable and dependent on feedstock type and pyrolysis method. This variability affects analytical reproducibility and method validation. Additionally, many extraction protocols still require the use of harmful solvents during the desorption phase, clashing with the goals of green analytical chemistry. Development of efficient, environmentally friendly elution methods is thus imperative. Integration with modern and miniaturized sample preparation formats is often hindered by the absence of standardized formats, further limiting widespread adoption. Another critical issue is the lack of regulatory clarity and formal recognition concerning the use of biochar sorbents. These materials are not yet recognized within official validation frameworks or standard operating procedures for food analysis. Without standardized reference materials or interlaboratory validation efforts, regulatory acceptance remains unlikely.

5. Conclusions and future perspectives

This review has examined the current state of biochar-based sorbents in the context of food analysis, offering a systematic evaluation of their origins, functional characteristics, and analytical applications. Biochar, derived from renewable biomass, aligns well with the principles of GAC offering biodegradability, cost-effectiveness, and potential reusability. However, while its role in environmental and pharmaceutical fields is well established, its integration into food analysis workflows remains comparatively underdeveloped. This work reveals that biochars derived from fruit waste are the most extensively applied in food matrices, particularly in aqueous-based samples such as juices or water for human consumption. These sorbents demonstrate notable extraction efficiency and compatibility with miniaturized formats. Nut- and seed-based biochars offer high selectivity, especially when modified, but their applicability across complex food matrices is still limited. Cereal- and wood-based biochars show promise in terms of structural stability and extraction reproducibility, yet their solvent demands and matrix limitations must be addressed. From an analytical chemistry standpoint, the most frequently studied analytes include pesticides, PAHs, and heavy metals, reflecting key food safety concerns. Aqueous matrices such as drinking water, fruit juices, and milk dominate the current research landscape, with lipid-rich or complex solid matrices being underrepresented.

Additionally, it is evident that there is still significant room for improvement in the analytical workflow involving biochar-based sorbents. One of the primary priorities for future research is the implementation of greener and more sustainable procedures that align with the principles of GAC. In particular, the widespread use of conventional organic solvents poses serious environmental and health concerns. These solvents are typically classified as non-recommended due to their toxicity and poor biodegradability. Therefore, future studies should actively explore the use of green solvents, including bio-based solvents, ionic liquids, and deep eutectic solvents, to minimize solvent consumption and environmental burden.

Another key direction is the improvement of biochar reusability. Currently, many biochar-based sorbents suffer from irreversible adsorption of analytes or exhibit limited regeneration efficiency, which restricts their use in repetitive analytical cycles. Strategies such as surface modification with reversible functional groups, development of thermal or solvent regeneration protocols, and incorporation of magnetic properties to facilitate recovery should be explored. Such improvements would not only reduce operational costs but also contribute

to waste minimization. Enhancing matrix compatibility also remains a major challenge, especially for complex and heterogeneous food samples such as dairy products, emulsions, and fat-rich matrices. Tailoring biochar physicochemical properties to improve selectivity and reduce matrix interferences will be crucial for broadening their applicability. For instance, controlling porosity, surface polarity, and pH-dependent functionalities could help improve performance across a wider range of food types.

From a materials innovation perspective, future research should focus on fine-tuning biochar properties through advanced functionalization strategies. These may include doping with heteroatoms, embedding nanomaterials (e.g., metal-organic frameworks, carbon dots, graphene derivatives), or grafting molecular recognition elements such as aptamers or MIPs. Such modifications could enhance adsorption efficiency, target specificity, and selectivity, expanding the analytical scope to include emerging contaminants and trace-level analytes.

Moreover, the integration of biochar-based sorbents into advanced analytical platforms also represents a promising avenue. Coupling with miniaturized sample preparation techniques could allow for higher throughput, automation, and lower solvent usage. Similarly, pairing with sensitive detection systems, including LC-MS/MS or high-resolution mass spectrometry, could enhance analytical reliability and sensitivity in complex matrices. Finally, regulatory and standardization aspects must be addressed to enable widespread adoption of biochar in routine food analysis. Currently, there is a lack of inclusion of biochar-based sorbents in official validation frameworks, certified reference materials, and standard operating procedures. Efforts should be made to generate robust validation data, promote interlaboratory studies, and establish quality control guidelines that ensure reproducibility and regulatory compliance.

CRedit authorship contribution statement

Carla Iglesias-Martín: Writing – original draft, Visualization, Investigation, Formal analysis, Data curation. **Ana M. Ares:** Writing – review & editing, Conceptualization. **José Bernal:** Writing – review & editing, Conceptualization. **Adrián Fuente-Ballesteros:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Abbreviations

ACN	acetonitrile
AGREE	analytical GREENness metric
AGREEprep	analytical greenness metric for sample preparation
BAGI	blue applicability grade index
BET	Brunauer–Emmett–Teller
BJH	Barrett–Joyner–Halenda
ComplexGAPI	green analytical procedure index
DAD	diode–array detector
DCM	dichloromethane
D-μ-SPE	dispersive micro–solid phase extraction
DLLE	dispersive liquid–liquid extraction
DSPE	dispersive solid phase extraction

EA	elemental analysis
EDX/EDS	energy-dispersive X-ray spectroscopy
ESI	electrospray ionization
FAAS	flame atomic absorption spectrometry
FPSE	fabric phase sorptive extraction
FTIR	Fourier-transform infrared spectroscopy
GAC	green analytical chemistry
GC-ECD	gas chromatography–electron capture detection
GC-FID	gas chromatography–flame ionization detection
GC-MS	gas chromatography–mass spectrometry
GC-MS/MS	gas chromatography–tandem mass spectrometry
HPLC	high performance liquid chromatography
IT-SPME	in-tube solid-phase microextraction
LOD	limits of detection
LOQ	limit of quantification
MeOH	methanol
MIP	molecularly imprinted polymer
MIP-SPE	molecular imprinted polymer-based solid phase extraction
MIPMs	molecularly imprinted polymer microspheres
MIPs-DSPE	molecularly imprinted polymer for dispersive solid-phase extraction
MOF	metal-organic framework
MSPE	magnetic solid-phase extraction
MSPD	matrix solid-phase dispersion
μ-SPE	micro-solid phase extraction
PAHs	polycyclic aromatic hydrocarbons
PDA	photodiode array detection
PT-μ-SPE	pipette-tip micro solid-phase extraction
PT-SPE	pipette tip-solid phase extraction
QuEChERS	quick, easy, cheap, effective, rugged, and safe
RAPI	red analytical performance index
RDSE	rotating-disk sorptive extraction
SEM	scanning electron microscopy
SPE	solid-phase extraction
TEM	transmission electron microscopy
TFME	thin-film microextraction
UHPLC-MS/MS	ultra-high-performance liquid chromatography tandem mass spectrometry
UHPLC-Q-TOF/MS	ultra-high-performance liquid chromatography coupled with quadrupole time-of-flight tandem mass spectrometry
UV-Vis	ultraviolet-visible detection
VIGI	violet innovation grade index
VSM	vibrating sample magnetometry
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

Data availability

Data will be made available on request.

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