Review Article

Recent applications of chromatography for determining microplastics and related compounds (bisphenols and phthalate esters) in food

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ARTICLE INFO	ABSTRACT
	The presence of microplastics in food poses a significant concern due to its potential impact on human health. Numerous studies have focused on spectroscopic techniques to identify and confirm those contaminants in food, but less attention has been paid to chromatographic techniques. Thus, this article provides a comprehensive review of the recent applications of chromatographic techniques in the analysis of microplastics and related compounds such as bisphenols and phthalate esters in food from 2018 to 2022. The study covers and discusses a range of different chromatographic applications (gas and liquid chromatography). Furthermore, this review also explores different sample treatments employed for extracting these compounds from food matrices, while offering a detailed analysis of their principles, advantages, and disadvantages. This review provides readers with an overview and valuable insights into the analytical performance and potential applications of these chromatographic techniques in the analysis of microplastics and related compounds in food.

1. Introduction

Plastics are widely used, owing to their ease of production, light weight, stability, versatility, and insulation properties. Despite global efforts to improve plastic recycling, improper disposal of plastics remains a prevalent global trend. This improper disposal is the primary cause of unregulated plastic release into the environment. Once plastic particles enter the environment, they undergo weathering and fragmentation due to external factors, such as the mechanical forces of water, ultraviolet radiation, and biological metabolism. These factors

lead to the formation of smaller plastic particles known as microplastics (MPs; [1]). In recent years, there has been increasing awareness and concern regarding MPs. These small plastic particles, ranging in size from 1 μm to 5 mm, have garnered significant attention, since their ability to enter different environmental matrices make them a complex challenge to address. This widespread distribution of MPs has led them to become *contaminants of emerging concern* [1–3] because of their potential tissue penetration and adverse effects resulting from their small sizes [4]. Until recently, analysis of MPs has primarily focused on aquatic environments. However, food products are also susceptible to

microplastic contamination. Contamination can occur through various pathways, including the presence of MPs in the soil and in plants. Furthermore, MPs can be introduced during food processing and packaging, posing an additional source of contamination [5]. Toxicological studies on MPs are increasing rapidly. Experiments show that the exposure to microplastics induces a variety of toxic effects, including oxidative stress, metabolic disorder, immune response, neurotoxicity, as well as reproductive and developmental toxicity [1,6–9].

However, it should be noted that the toxicity of MPs is due not only to their own nature but also to the additives used in their manufacture to give them certain properties, as for example to enhance their durability, flexibility, and temperature resistance. The most commonly used can be classified as: i) chemical agents (flame retardants, plasticizers, antioxidants, UV stabilizers and pigments); ii) chemicals from UV degradation of plastic (bisphenols (BPs) and phthalate esters (PAEs)); iii) chemical products derived from the recycling of plastic waste; iv) heavy metals [10,11]. On the other hand, MPs can act by adsorbing other compounds within their structures, such as persistent organic pollutants, polycyclic aromatic hydrocarbons, dichlorodiphenyltrichloroethane and heavy metals that cause metabolic changes and biochemical disturbances inducing ecotoxic responses. Furthermore, the dangers imposed by such chemicals adsorbed on the surface of MPs are greater than those directly emitted by plastics [12]. Among the most used additives that can be found in MPs, it is worth highlighting BPs and PAEs. These compounds have been called "everywhere chemicals" due to their widespread use in the manufacture and processing of plastic and resin and their ubiquity in the human body, wildlife, and the environment [13]. Thus, it is not surprising that they have been simultaneously investigated in several publications and related to the presence of MPs [13–15].

PAEs are a class of esters formed from phthalic acid and alcohols with 4–15 carbon atoms [16,17] (see structures of some PAEs in Supplementary Material, Fig. S1). PAEs rely solely on physical bonds, instead of covalent bonds that are strongest, with plastic substrates. This characteristic makes them prone to migration from plastic packaging to food items during the production and storage processes, which can eventually pose a risk to human health. In fact, studies have shown that prolonged consumption of PAEs through food intake can have detrimental effects on human well-being. These effects include disruption of endocrine functions, reproductive toxicity, the potential for birth defects, and the development of cancerous conditions [18]. Owing to their harmful effects, the use of PAEs have been restricted in food contact materials, as outlined by the European Commission Regulation (EU 10/2011). The specific migration limits of dibutyl phthalate (DBP), di(2-ethylhexyl) phthalate (DEHP), benzyl butyl phthalate (BBP), and the sum of diisononyl phthalate (DINP) and diisodecyl phthalate (DIDP) are 0.3, 1.5, 30, and 9 mg kg^{-1}, respectively [19]. Therefore, it is crucial to develop efficient methods to detect and quantify the presence of PAEs in food products with the aim of guaranteeing safety for humans.

BPs are organic compounds characterized by a structure comprising of two phenol rings (see structures of some BPs in Supplementary Material, Fig. S2). These rings possess hydroxy groups at the *para* positions and are connected by either a carbon or sulphur bridge, depending on the specific analogue [20]. The most well-known bisphenol is bisphenol A (BPA), a monomer used in the production of polycarbonate (PC) plastics and epoxy resins, which are commonly employed in the manufacture of food coatings and plastic food containers [21]. BPA is widely recognized as a hazardous substance due to its endocrine disrupting properties. Therefore, its concentration in foodstuffs is regulated within specific migration limits. The European Union Commission has established a migration limit of 0.05 mg kg^{-1} of food for BPA from coatings onto food, and its usage is prohibited in products intended for infants and young children. A wide range of analogues of BPA exists, including bisphenol B (BPB), bisphenol C (BPC), bisphenol S (BPS), and more. Additionally, there are derivatives, such as bisphenol A diglycidyl ethers (BADGEs) and bisphenol F diglycidyl ethers (BFDGEs). However, migration limits for BPA analogues, except for BPS, set at 0.05 mg kg^{-1},

have not yet been established [22]. Consequently, these analogues have been used as substitutes.

It should be mentioned that BPs and PAEs have been classified as endocrine disruptors (EDCs), which are exogenous synthetic or natural substances that can affect virtually any aspect of the endocrine system as well as physiological functioning. Although the effects of EDCs on human health are not yet fully understood, their effects on women are especially serious. BPA is known to have structural similarity to some hormones, especially those that control breast development. Therefore, it is possible that its exposure leads to proliferation, giving rise to cancer cells in the breast tissue [23–25]. Exposure to BPA and PAEs during pregnancy can cause problems in the correct neuronal development of the foetus, risk of diabetes, development of heart disease [26] and, in the case of the mother, development of thyroid cancer [27] and even produce spontaneous abortions [28,29]. Notably, many endocrine disruptors, such as MPs, PAEs, and BPs, can act synergistically to produce additive effects in the human body [30]. The analysis of these compounds in any food matrix can be challenging for two primary reasons. In the first place, the complexity of food matrices, which may contain interfering compounds, can produce important matrix effects. Furthermore, MPs, BPs and PAEs are ubiquitous in the laboratory, making it crucial to minimize background contamination to ensure accurate analysis of samples.

In recent years, there has been a notable increase in studies focusing on the chromatographic determination of MPs, BPs, and PAEs in various food matrices (see Fig. 1A). However, as can be seen, the number of publications related to additives (BPs and PAEs) was much higher than that related to MPs. As can be expected, several review articles have been published dedicated to commenting on the most relevant works related to the determination of MPs and related products (BPs and PAEs) in food. For example, two recent publications [16,31] discussed the analysis of MPs in food, highlighting the predominant use of spectroscopic techniques, while briefly mentioning chromatographic methods. However, the coverage of chromatographic techniques is not as detailed, and there is a lack of discussion on methods for additives and other compounds associated with MPs. In one of these works [31], several recent publications are reviewed, focusing on various aspects such as extraction, separation, and detection. In addition, quality assurance/quality control measures implemented for the analysis of MPs have been also discussed. While those studies primarily focus on articles related to MP analysis, there is limited discussion on chromatographic methods. Finally, two additional publications deserve attention, focusing on the most significant applications of spectroscopic techniques [7] and mass spectrometry [32] for the analysis of MPs in food. These works provide more in-depth insights into the chosen techniques, particularly in the case of the mass spectrometry article [32], which discusses various chromatographic applications based on GC and LC. However, it is important to note that these publications exclusively focus on MPs, with no inclusion of other compounds. To find review papers discussing methods for determining BPs and PAEs, it is necessary to search for specific papers.

There are limited publications focusing on methods for simultaneously determining multiple BPs. However, a recent paper by Ali et al. [33] provides a compilation of various recent studies, emphasising the extraction and subsequent analysis of BPs using LC with different detectors. The paper discusses several extraction techniques such as solid-phase extraction (SPE), magnetic-SPE (MSPE), and dispersive liquid-–liquid microextraction (DLLME). It is important to note that the discussed articles exclusively focus on BPs and are not limited to food analysis. Furthermore, it is worth mentioning that among the BPs, BPA has been extensively studied, leading to numerous publications exclusively dedicated to this compound. One particularly comprehensive review article describes different sample treatments employed to assess the migration of BPA and its analogues from packaging materials to food and beverages [34]. It is important not to overlook a valuable article [35], despite not being the most recent, as it serves as an excellent
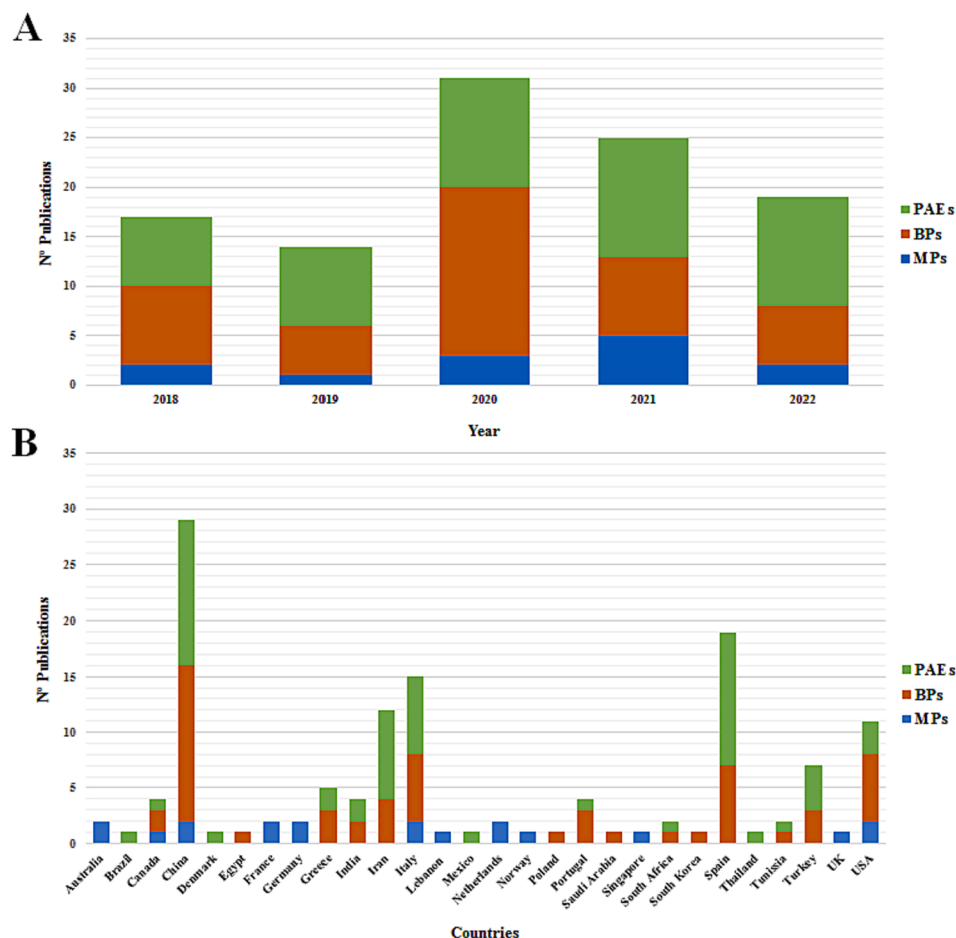
Fig. 1. Evolution of the number of published works **A)** per year and **B)** per country of origin in the last five years (2018–2022) related to the chromatographic determination of microplastics (MPs), bisphenols (BPs) and phthalate esters (PAEs) in food. Bibliometric analysis is described in *Section 2.1*.

starting point for studying BPA in food. This comprehensive resource summarizes the most commonly used analysis methods for determining BPA in food, highlighting prevalent sample treatments such as solvent extraction and SPE as well as emphasising separation techniques, with LC and various detectors being prominent.

In the case of PAE analysis, several review articles have been published, assessing the analytical methods employed for their determination in food [36–38]. The most prevalent technique employed is GC, particularly in conjunction with MS. It should be mentioned that a dedicated review exclusively focused on the GC–MS coupling for PAE analysis has been published [38]. This review discusses various extraction techniques, including solvent extraction, solid-phase microextraction (SPME), DLLME, microwave-assisted extraction (MAE), and more. Similarly, these extraction, separation, and detection techniques are discussed in conjunction with others in more general articles on the analysis of PAEs in food. The difference lies in the specific time frame of recent article discussions in the first review [37], while the second review provides a broader perspective by commenting on older articles [36].

Thus, it is clear, considering the numerous existing publications, that the determination of MPs, BPs and PAEs in food has attracted the attention of researchers in recent years, mainly using spectroscopic methods for MPs, and chromatographic methods for the other compounds. However, nowadays, chromatographic methods are gaining interest for the quantification of MPs, but as to date and to our knowledge there is no work where some of the main and most recent chromatographic applications in food analysis for determining MPs together with some of the additives/compounds that are associated with their presence (BPs and PAEs) are discussed together. As mentioned

previously, BPs and PAEs have been chosen among other compounds related to MPs because their study is attracting the attention of many researchers in recent years due to their ubiquity and their potential adverse effects on health (EDCs). This has meant that they have been studied together in several works, in which chromatography has been used for their determination. By examining the latest research in this field (2018–2022), we aimed to provide a comprehensive overview of the recent applications of chromatography for assessing the presence of MPs and related compounds (BPs and PAEs) in food, with the added advantage over previous articles of collecting all the relevant information in a single article.

Summarising, it is true that in recent years several papers have been published related to the analysis of these compounds in various matrices, including food. However, with this review we focus on the simultaneous chromatographic analysis of these compounds in food in a single document, unlike in previous works. The work is laid out in accordance with the different families of compounds, indicating and discussing the most common sample treatments and chromatographic methods used to determine those compounds in food. Readers interested in more specific details, such as the occurrence of compounds (MPs, BPs and PAEs) and/or potential toxicities, fate, other methods of analysis or additives, can refer to the some of the above-mentioned reviews and to the related literature.

2. Recent chromatographic applications in food analysis

Firstly, we carried out a small bibliometric study to show the impact and influence of the topic discussed in this review within the scientific

community. Afterwards, and considering the differences in the characteristics and analysis requirements of MPs, BPs, and PAEs, individual sections will be included for each of them, and in the case of BPs and PAEs as they have been studied jointly in some cases, a section will be included where these works will be discussed.

2.1. Bibliometric analysis

To deliver a comprehensive review, it is of utmost importance to perform scientometric evaluation of the available literature on chromatographic determination of MPs, BPs and PAEs in the selected period (2018–2022; data up to November 2023). The methodological approach employed involved bibliometrically tracking scientific articles from Clarivate's Web of Science™ Core Collection (WOS) database [39]. The search has been done using keywords [(chromatography) and/or (microplastics) and/or (bisphenols) and/or (phthalate esters) and/or (food) and/or (plastic additives) and/or (beverages) and/or (extraction) and/or (isolation) and/or (quantification) and/or (separation) and/or (determination) and/or (analysis)] among several others. The acquired results were limited to original papers written in English and published. In total 119 papers (up to 2022) were recorded and all their abstracts were read to confirm their relevance towards the review topic. Based on these finding, the following interesting facts were identified.

The total number of papers found specifically related to the analysis of MPs, BPs and PAEs in food was 119, of which 15 (~13 %) were reviews and 104 (87 %) research papers. There was a significant increase in the number of publications until 2020, which more than doubled in 2019, although the number of publications has decreased in the following years (see Fig. 1**A**). There are fewer publications devoted to

the chromatographic determination of MPs in relation to BPs and PAEs, which will be explained in *Section 2.2*. It should also be noted that as the topic of study is of global interest, the nationality of the researchers who have carried out research is diverse and broad, with more than 25 different countries covering the five continents, in particular, China (>25), followed by Spain and Italy (see Fig. 1**B**). In relation to the areas of research, of the total eight categories, Food Science & Technology – Science Citation Index Expanded (SCIE) was the highest number of publications (54) representing 45 % of total papers, followed by Chemistry, Analytical - SCIE with 38 publications (32 % of total). The eight categories of journal areas and their publications count are described in Table 1. The prevalence of the categories mentioned above makes sense considering the topic of this review. Most publications are dedicated either to commenting on the results of the analysis of these compounds in foods along with the development of new analysis methods; hence, the high number of articles in Analytical Chemistry, and Food Science & Technology. Also, in many cases the importance of these compounds as contaminants has been considered and they have been studied together with other environmental matrices, which implies that the area of Environmental Sciences has a high number of publications. In addition to the areas, it is also interesting to note that the articles have been published in 57 different journals, although most of them are concentrated in two journals related to food science and technology (Food Chemistry and Food Analytical Methods), followed by three journals in analytical chemistry (Journal of Chromatography A, Microchemical Journal and Analytical and Bioanalytical Chemistry). These results correspond perfectly with what was mentioned above regarding scientific areas/fields. The main ten journals with the highest number of publications, their number of papers and impact factor are listed in Table 2. It is interesting to highlight the high impact factor of many of these publications, which places them in the first quartile of their areas, which highlights the relevance and interest of the topic discussed in this review. To conclude the bibliometric study, it can be highlighted that although the publications correspond to a recent period (2018–2022), there are already three of them that exceed 100 citations, and in all cases, they refer to the analysis of MPs by GC–MS, which once again highlights the interest that the topic under study in this work attracts. The ten publications with the highest number of citations are summarised in Table S1 (see Supplementary Material). However, no author or research group can be highlighted since there are not many repetitions in the list of publications.

To summarise, it can be concluded that the chromatographic analysis of MPs, BPs and PAEs is a topic that attracts the attention of researchers around the world and encompasses various areas/fields of knowledge. This has been demonstrated not only by the number and origin of the

Table 1

Ranking main areas/field research of papers published related MPs, BPs, and PAEs analysis in food. Bibliometric analysis is described in *Section 2.1*.

Ranking	Subject area	N° Publications (%Total)*
1	Food Science & Technology - SCIE	54 (45)
2	Chemistry, Analytical - SCIE	38 (32)
3	Chemistry, Applied – SCIE	27 (23)
4	Environmental Sciences - SCIE	18 (15)
5	Chemistry, Multidisciplinary – SCIE	5 (4)
6	Toxicology – ESCI	1 (1)
7	Polymer Science - SCIE	1 (1)
8	Nutrition & Dietetics – SCIE	1 (1)
9	Not Indexed	5 (4)

*Some publications are indexed in two different categories.
SCIE, Science Citation Index Expanded.
ESCI, Emerging Sources Citation Index.

Table 2

The ten top most productive journals related to the chromatographic determination of MPs, BPs and PAEs and their impact factor range. Bibliometric analysis is described in *Section 2.1*.

Ranking	Journal	Subject Area	N° Publications (% Total)	Impact factor * (Quartile)
1	Food Chemistry	Food Science & Technology – SCIE; Chemistry, Applied – SCIE	20 (17)	8.8 (Q1)
2	Food Analytical Methods	Food Science & Technology – SCIE;	13 (11)	2.9 (Q3)
3	Journal of Chromatography A	Chemistry, Analytical - SCIE	7 (6)	4.2 (Q2)
4	Microchemical Journal	Chemistry, Analytical - SCIE	5 (4)	4.8 (Q1)
5	Analytical and Bioanalytical Chemistry	Chemistry, Analytical - SCIE	5 (4)	4.3 (Q1)
6	International Journal of Environmental Research and Public Health	Environmental Sciences - SCIE	4 (3)	4.6 (Q2)[2021]
7	Food Additives and Contaminants- Part A	Food Science & Technology – SCIE; Chemistry, Applied – SCIE	4 (3)	2.9 (Q2)
8	Journal of Separation Science	Chemistry, Analytical - SCIE	3 (3)	3.1 (Q2)
9	Journal of Agricultural and Food Chemistry	Food Science & Technology – SCIE; Chemistry, Applied – SCIE	2 (2)	6.1 (Q1)
10	TRAC-Trends in Analytical Chemistry	Chemistry, Analytical - SCIE	2 (2)	13.1 (Q1)

* Impact factor was taken from JCR 2022.

publications, but also by the quality of the journals in which these studies have been published, and the number of citations that some of them have achieved in such a short period of time.

2.2. Determination of MPs in food

Although tremendous efforts have been made in the last decade to identify MPs in food, standardized experimental protocols have not been developed [5]. While numerous studies have focused on spectroscopic techniques to identify and confirm MPs in food [7,31,40], there has traditionally been limited emphasis on quantitative analysis. It is important to consider that traditional chromatographic techniques such as gas chromatography (GC) or liquid chromatography (LC) are not the preferred options because MPs have high molecular weights and limited solubility in most solvents. In fact, many studies do not aim to quantify MPs, but rather focus on their detection and characterization using microscopic and spectroscopic techniques, such as Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy. Although these techniques are easy to perform and provide valuable information regarding the number, chemical composition, shape, and size distribution of MPs, they only yield an approximate estimation of their quantities. More recently imaging techniques have become popular, which enable an almost automatic scanning of prepared samples for characteristic absorption bands of selected polymers. However, this technique is time-consuming because analysis time increases substantially with decreasing particle size [41]. If the objective is precise quantification, alternative approaches combining thermal decomposition with mass spectrometry are more suitable [3,42]. The principle of these methods is that polymers undergo thermal decomposition through different mechanisms, producing specific products with related signal intensities, indicator ions, and retention times, which enables the detection and quantification of several polymers in one run. A promising and increasingly employed technique in this context is gas chromatography–mass spectrometry with pyrolysis (Py–GC–MS). This technique uses heat in an inert environment to thermally decompose polymeric materials in a predictable manner. The resulting pyrolysis products are subsequently separated using GC based on their size and polarity, providing a chromatogram with mass spectrometry data throughout the chromatogram. These data can be compared with a known reference library to identify the specific class of polymers being analyzed [43]. It is crucial to consider that pyrolysis temperature can significantly affect the generated pyrolysis products, which may differ from those found in the reference library. Because many libraries are based on pyrolysis at 600 °C, this can pose challenges for the identification of polymers at different temperatures. However, by considering this factor, it is possible to create a customized database with other pyrolysis temperatures that enables accurate polymer identification [44]. Nevertheless, Py–GC–MS has certain limitations. These include the requirement of a limited sample mass, the challenge of manually transferring hand-picked particles into the pyrolysis cup, the lengthy analysis time, and its destructive nature [43]. In addition, Py–GC–MS does not provide information on the colour, shape, or size of MPs, and has shown a lower sensitivity for polar polymers, which can be overcome by applying thermally assisted hydrolysis or methylation prior to analysis [31]. However, this technique can be used in conjunction with microscopic and spectroscopic techniques to provide this type of information [31,45]. Despite these limitations, a key advantage of Py–GC–MS is the selectivity of mass spectrometry, which enables simplification of sample treatment in many cases. Pyrolysis is usually performed online; however, in some cases off-line pyrolysis is employed as an alternative technique. Nevertheless, it has been principally employed for analysing environmental samples [17] which explains the limited number of studies in which it was used for the analysis of MPs in food. Thermogravimetric analysis (TGA) could be also employed for determining MPs in food. The principle of TGA differs from pyrolysis, as it measures the weight loss of a sample as it is heated at a programmed rate in a controlled gaseous environment. TGA–FTIR/GC–MS is a viable technique, since it also allows for the examination of thermal products through mass spectrometry and enables quantitative analysis. Indeed, TGA–MS has been reported in the literature as an easy, fast, and valid screening method for the detection of MPs [31]. Despite being capable of detecting and characterizing different MPs, TGA–MS is not a competitive quantitative method in terms of accuracy, presumably because transfer capillaries get easily clogged by plastic degradation products [46], and the presence in the same sample of different polymers having similar degradation temperature, makes results interpretation particularly [47]. However, the use of TGA–FTIR–GC–MS has solved some of the above-mentioned problems, although it has increased the complexity of the method development [31].

As shown in Table S2 (see Supplementary Material), thermal methods were employed for the quantification of MPs in all studies except one [48] in which LC–MS/MS (triple quadrupole-QqQ) was used (see Fig. 2A). A hydrophilic interaction liquid chromatography (HILIC) silica column was applied to detect some of the polyamide (PA) 6 monomers in gradient elution mode, and a C_{18} column was selected to determine PA 66 monomer by using isocratic conditions (see Fig. 3A). HILIC eliminates much of the cost and labour for polar analysis, compared to other techniques such as derivatization, ion exchange, ion pairing, and normal-phase LC, but conversely, method development and troubleshooting can pose a challenge when developing HILIC approaches. However, complex sample treatment is required, including an acid depolymerization followed by a solvent extraction or a solid-phase extraction (SPE) clean-up for determining nylon MPs. It should be specified that depolymerization is characterized by the breaking of polymeric bonds, and by assuming that a polymer can be completely depolymerized, its mass can be calculated by using the emerging functional monomer compounds, as it was done in the present study. By using the proposed method, nylon MPs can be quantified without separation from the samples, which increases the efficiency of analysis and prevents the loss of MPs or incidence of error caused by separation. Furthermore, it is worth noting that the analyzed compound in that work was exclusively PA, which can also be analyzed by Py–GC–MS. Therefore, thermal methods combined with chromatography remain the best approach for quantitative analysis of MPs, as previously discussed (see Fig. 2A). Py–GC–MS (quadrupole-SQ) was employed in most of the works included in Table S2 (see Supplementary Material; [2,3,42–44,49–51]). It should be mentioned that helium (carrier gas) and temperature programs were used in all cases. In relation to the columns, non/ultra-low-polar stationary phases have been predominantly employed (UA5(MS/HT) [2,3,42,43], TG-5SilMS [31]; Rxi-5MS [44,49], HP-5MS [50,51], DB-5MS [52], Rxi-5SilMS [53]; while, only in one case [54], an intermediate polarity column (ZB-35) was employed (see Fig. 3B and Table S3, Supplementary Material). Precisely, in this latter study, off-line pyrolysis was used to quantify polystyrene (PS) in mussels that were exposed to microparticles. The highest value of PS recorded was 14 µg g^{-1}. The off-line technique can be a useful approach in situations where a Py–GC system is not available. While off-line pyrolysis allows for the use of a higher amount of sample, which results in enhanced sensitivity, it is important to consider that this technique involves additional procedural steps compared to direct Py–GC–MS. These extra steps increase the risk of contamination, which is a significant limitation, especially in trace analysis. In the above-mentioned study, the contamination risk resulted in a relatively high limit of quantification (LOQ) of approximately 1 mg kg^{-1}. It is worth noting that some studies used Py–GC–MS/MS (QqQ; TG-5SilMS [45]; RXi-5SilMS [53]), which enhances the detection performance, thus permitting a simpler sample preparation, although the cost is higher. In these studies, sample treatment consisted of digestion. For example, Albignac et al. [45] implemented a straightforward sample preparation method involving potassium hydroxide digestion, which yielded extraction procedure efficiencies ranging from 82 to 129 % for the six studied polymers (PC, polypropylene-PP, polyethylene-PE, polyethylene terephthalate-PET,
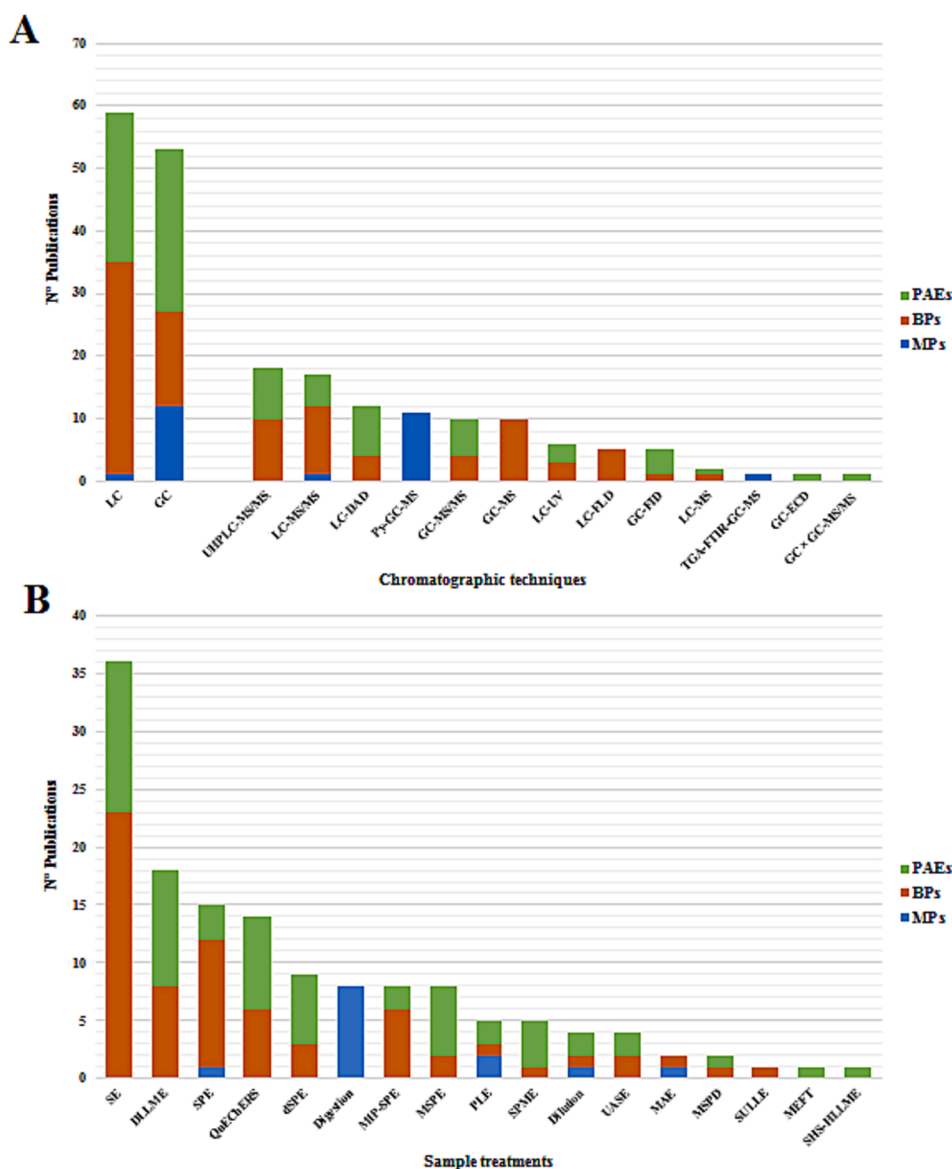
Fig. 2. Summary of the **A)** chromatographic techniques and **B)** sample treatments used in the last five years (2018–2022) to determine microplastics (MPs), bisphenols (BPs) and phthalate esters (PAEs) in food. Bibliometric analysis is described in Section 2.1. **DAD,** diode array detector; **DLLME,** dispersive liquid–liquid microextraction; **dSPE,** dispersive SPE; **ECD,** electron capture detector; **FID,** flame ionization detector; **FLD,** fluorescence detector; **FTIR,** thermal gravimetric analysis; **GC,** gas chromatography; **GC × GC,** comprehensive GC; **LC,** liquid chromatography; **MAE,** microwave assisted extraction; **MEFT,** magnetic effervescent tablet-based microextraction; **MIP,** molecularly imprinted polymers; **MS,** mass spectrometry; **MS/MS,** tandem mass spectrometry; **MSPD,** matrix solid-phase dispersion; **PLE,** pressurized liquid extraction; **Py-GC–MS,** pyrolysis-GC–MS; **QuEChERS,** quick, easy, cheap, effective, rugged, and safe; **SE,** solvent extraction; **SHS-HLLME,** liquid–liquid microextraction with switchable hydrophilicity solvent as extraction phase; **SPE,** solid-phase extraction; **SPME,** solid-phase microextraction; **SULLE,** sugaring-out assisted liquid–liquid extraction; **TGA,** thermal gravimetric analysis; **UASE,** ultrasound assisted extraction.

poly(methyl methacrylate)-PMMA, PS). The total polymer content obtained varied between 105 and 7780 mg kg^{-1}. There is only one work in which thermal gravimetric analysis–Fourier-transform infrared spectroscopy–GC–MS (TGA–FTIR–GC–MS; DB-5MS, SQ) was employed [52] to accurately quantify MPs in mussels. The results revealed an average plastic content of 0.58 mg kg^{-1} of tissue, with a range of 0.16 to 1.71 mg kg^{-1}.

As mentioned previously, the employed techniques facilitate a straightforward sample preparation process, even for foods. However, it is crucial to be careful during sample treatment to minimize contamination from plastic particles present in the laboratory environment. Many different methods of mitigation of the cross-contamination of MPs exist. A large majority are universal and can be applied irrespective of the type of analysed samples. The most popular methods include, among others: wearing clothing containing no plastic fibres, cleaning

laboratory surfaces and entire equipment before use, covering samples and laboratory equipment with aluminium foil, work in conditions of controlled air flow, and the use of an exclusively glass or metal laboratory [55]. More extreme, but effective, measures to remove contamination involve calcination of the stainless steel and glass materials at high temperatures (>450 °C), and cleaning with previously filtered solvents [45,56]. The effectiveness of cleaning procedures can be cursorily checked by examining tools, materials, and equipment under a stereomicroscope; while it is essential to analyse procedural blanks between each set of samples, to control/detect potential contamination [57]. Such analysis of procedural blanks is based on the performance of an additional replicate of the methodology, using the same reagents and amounts but without the sample matrix. That constitutes the most relevant strategy to evaluate the degree of contamination during the analytical method developed, and should be done in parallel with the
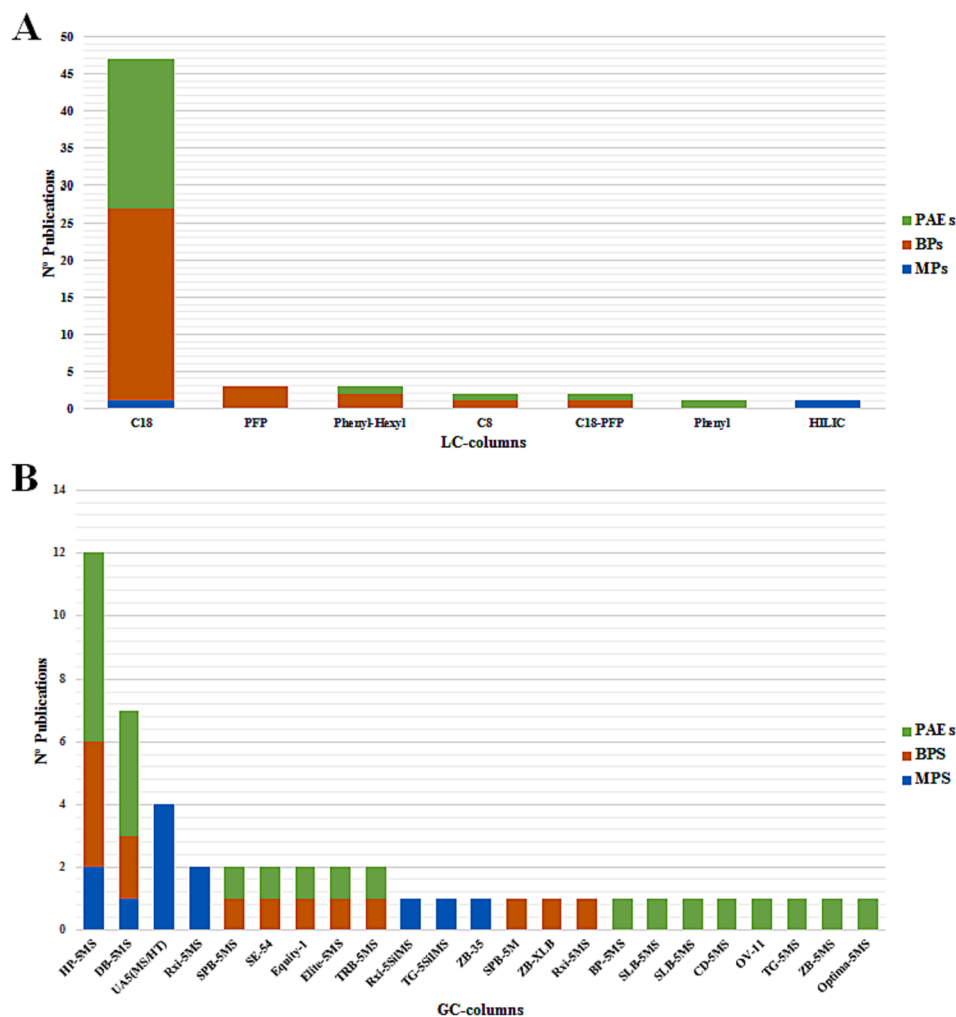
Fig. 3. Summary of the **A)** HPLC and **B)** GC columns employed in the last five years (2018–2022) for determining microplastics (MPs), bisphenols (BPs) and phthalate esters (PAEs) in food. Bibliometric analysis is described in *Section 2.1*. **HILIC**, hydrophilic interaction chromatography; **PFP,** pentafluorophenyl.

analysed samples [36]. Various sample treatments can be used for the analysis of MPs, including digestion, pressurised liquid extraction (PLE), and microwave-assisted extraction (MAE; see Fig. 2B). In PLE, the extraction process is carried out at elevated pressures to work with solvents in the liquid phase at temperatures above their boiling point. The simultaneous use of high pressures and temperatures improves the solubility and mass transfer properties of the target compounds, and leads to faster extraction processes than classical procedures (e.g., Soxhlet), using lower volumes of solvents and thus reducing sample dilution. Meanwhile, MAE is based on the principle of different boiling points of liquids under different air pressures, and the combined microwave extraction and vacuum technology. It is an automated technique that offers several benefits. It significantly reduces extraction time, solvent usage, and energy consumption. Furthermore, it enables multiple simultaneous extractions, thereby increasing the daily sample processing capacity. In fact, PLE and MAE are recognised as green extraction techniques due to their low consumption of organic solvents, although the cost and complexity of the methodologies will increase. Among these treatments, digestion is the most used method in the analysis of MPs, with eight out of thirteen studies employing this technique ([3,43–45,49,52–54]), and in most cases, potassium hydroxide was used as solvent for performing the digestions. The removal of the organic matrix by enzymatic, oxidative, or acidic/basic digestion offers many advantages, as it is very efficient and inexpensive. Nevertheless, it is important to consider that the use of high temperature during digestion can potentially alter some polymers. For example, PET fibres are not

resistant to elevated temperatures such as 60 °C, which can lead to low recoveries [43]. Therefore, it is crucial to carefully select the optimal digestion temperature and the reagents based on the analyzed compounds to ensure good recoveries. The digestion process requires a considerable amount of time, usually overnight. By contrast, as was previously mentioned, MAE and PLE appear to be promising techniques for extracting MPs from samples in a more efficient and timely manner. These methods generally result in good recoveries ranging from 92.9 % to 119.7 % in one study [50] and from 78 to 100 % in another [43]. However, it is important to note that PET still presents problems as it has shown low recoveries in a number of cases. In all cases, MPs were detected in the samples. The polymer content varied across different types of polymers and between individual samples. For example, in the case of table salts [3], the total mass content of seven types of plastics ranged from 30 to 530 $\mu g\ kg^{-1}$. On the other hand, in store-bought rice, the average plastic concentration was 283 $\mu g\ g^{-1}$, indicating a substantial difference compared to the salt samples.

To sum up, according to the bibliography consulted, the best option for the chromatographic determination of MPs is the use of Py–GC–MS with non/low-polar stationary phases, helium as carrier gas and temperature programming. When using Py–GC–MS the sample treatments are not as complex as with other techniques, despite the complexity of the food matrices, and are generally based on a basic digestion with potassium hydroxide followed by filtration. This option is the simplest, but generally requires the use of large quantities of solvents and long times, so the use of PLE and MAE can serve as an alternative. They are

more expensive and complex, but they shorten the analysis time and reduce the solvent consumption. Now, we must not forget that in the analysis of MPs it is crucial to avoid contamination during all stages of the analytical process, and therefore contamination control measures must be taken to the extreme in the laboratory, and the analysis of procedural blanks must not be forgotten.

2.3. Determination of BPs in food

Current methods for determining BPs rely predominantly on chromatographic techniques such as LC with fluorescence (LC–FLD), ultraviolet (LC–UV), diode-array (LC–DAD), and MS detection (LC–MS or LC–MS/MS), as well as GC–MS, GC–MS/MS and GC with flame ionisation detector (FID; see Supplementary Material, **Table S4**). Ultra-high-performance liquid chromatography (UHPLC) has been selected in some studies [20,58–66], as this might obtain better resolutions and sensitivities, as well as shorter running times, implying lower solvent consumption, but the systems must be able to withstand the high pressures that will be required when using columns with such a small internal diameter.

Due to the low volatility of these compounds, GC analysis requires some pre-treatment derivatization steps such as alkylation, silylation, or acylation prior to chromatographic separation. These additional manipulations not only increase the analysis time but also diminish the reproducibility of the method and introduce a potential source of contamination [34].

A summary of appropriate publications can be found in **Table S4** (see Supplementary Material; [19,21,22,58–102]). As can be seen in Fig. 2**A**, LC is used more frequently than GC, as expected, to avoid the need for a derivatization step, while MS/MS (LC) and MS (GC) are the most employed detectors. It should be also remarked that reverse-phase liquid-chromatography with C_{18}-based columns and mobile phases mainly composed of water, acids, salts as aqueous constituents and methanol or acetonitrile as the organic solvents applied in gradient elution mode were mainly employed for determining BPs in food; while only in some cases, isocratic elution mode or other stationary phases like pentafluorophenyl (PFP; [72,89], C_8 [77], phenyl-hexyl [64,83], or even a mixed stationary phase PFP-C18 [95] were selected (see Fig. 3**A**).

LC coupled with optical techniques (FLD, UV, DAD) demonstrates great potential for the simultaneous determination of BPs, offering advantages such as simplicity, rapidity, high efficiency, and low cost. The absorption of BPs is often monitored at 225–230 nm, and the emission at 305–315 nm, depending on the BP analyzed. Indeed, DAD has usually been selected in articles in which only BPA was analyzed [74,82,92,95,99].

However, to enhance the selectivity and sensitivity when analysing BPs at low concentrations in complex matrices, MS/MS with a QqQ analyser is the preferred choice [20–22,58,59,61,63,64,66,69,70, 72,90,91,93]. Other MS analysers like triple quadrupole-linear accelerator trap mass QTRAP [65,89], which has the capability to function like a standard QqQ also doubles as a linear ion trap (LIT) for better specificity and quantitative performance, high resolution MS (HRMS) like quadrupole–time-of-flight (QTOF) [94] or Orbitrap [62], and simple SQ [79] have been selected in some studies. MS-based detectors provide better selectivity and sensitivity than optical ones, but it must also be considered that they are much more expensive and more complex to use. These compounds are usually analyzed in negative ionisation mode, producing $[M - H]^-$ ions, and BADGEs have a high tendency to form adducts in positive mode [20].

UHPLC–MS/MS (C_{18}, QqQ) was used, for example, to analyze eight BPs in eggs [60]. The LOQs of BPs ranged from 0.02 to 0.05 µg kg^{-1}, recoveries ranged between 82.7 % and 105.2 %, and the measured levels of BPs in the egg samples ranged from 0.28 to 15.3 µg kg^{-1}. In another study, HRMS (C_{18}, Orbitrap) was utilised, which has proven to be useful for the non-targeted analysis of a sample or targeted analysis of contaminant mixtures in a matrix. The LOQ of BPA was below 10 ng g^{-1}

in all commercial shellfish samples. Although HRMS was employed in this study because the analysis encompassed a wide range of contaminants alongside BPs, it is not essential when focusing solely on BP analysis.

Regarding the GC analysis of BPs in food, it should be commented that the experimental conditions are very similar to those mentioned for the analysis of MPs in foods. Helium (carrier gas), program temperatures, and non/ultra-low-polar stationary phases (ZB-XLB [68], SPB-5 [75], DB-5MS [76,80], HP-5MS [84,85,96,98], Rxi-5MS [87], SE-54 [97]; Equity 1 [98], SPB-5MS [100], Elite-5MS [101]; TRB-5MS [102]) were used in all cases except one, in which an intermediate polarity phase (Rxi-624Sil MS, fused silica) was employed [22] (see Fig. 3**B** and **Table S3**, Supplementary Material). Moreover, SQ has been the detector of choice when determining BPs by GC [22,68,75,76,80,96, 98,100,101] (see Supplementary Material, **Table S4**). GC–QqQ was only employed in two studies [84,85], as was ion trap (IT), another MS/MS detector [97,102]. Additionally, there is a study where GC-FID (HP-5MS) was used to quantify BPA in beverages [98]. The proposed method achieved a LOQ of 0.80 µg L^{-1}. The results showed a concentration range of BPA in the analyzed drinks ranging from 4.12 to 15.3 µg L^{-1}. However, it is worth noting that no further studies utilizing GC-FID have been found. It is likely that GC-FID was chosen in this study because the researchers were also analysing other compounds, such as PAEs.

Determination of BPs concentrations in food is a significant challenge, primarily because of the complexity of the matrix and the wide concentration ranges involved. For this reason, sample treatment is critical, and extensive and meticulous sample preparation protocols involving multiple steps are often necessary to isolate BPs from food samples, particularly when analysing solid matrices [79]. It is important to note that owing to the widespread presence of BPs in laboratory environments, it is crucial to take precautions to control the background values of the procedural blanks. Background contamination of BPs occurs at ng/L levels and mainly arises from solvents, SPE columns, glassware, plasticware and other reagents and laboratory tools. In general, heat-treated glassware (4 h at 400 °C) and solvent-washed materials are used as a precautionary measure to prevent background contamination [35]. BPs concentrations around 0.02 µg L^{-1} have been found in ultrapure water using highly sensitive methods; the contamination arises from the plastics used in the purification system and is removed by filtering the water through a hydrophobic membrane [35]. SPE cartridges have been known to cause BPA contamination at concentrations of around 0.04 µg L^{-1} [103]. The contamination was effectively removed by pre-washing the cartridges which at least 15 mL of methanol and was probably derived not from the sorbent material, but from the manufacturing process. Sample loading in SPE can introduce strong BPA contamination when glass syringes are utilized, mainly caused by the adhesive used to fix the needle. Contamination from SPE sample loading can be almost eliminated by replacing the vinyl tubes with Viton tubes from DuPont [35,103]. Finally, it is important that cleaning procedures are regularly checked as highlighted in the previous section for the determination of MPs in food.

The two most predominant techniques according to the revised literature used for sample treatment are solvent extraction (liquid–liquid extraction and solid–liquid extraction) and SPE, as shown in Fig. 2**B**. Solvent extraction shows good analytical performance and can be easily performed. Some solvents commonly used for the extraction of BPs are acetonitrile, hexane, ethyl acetate, methanol, and acetone. Although solvent extraction shows good analytical performance, its drawbacks, such as the formation of emulsions and the requirement of large volumes of organic solvents, make it a costly, time-consuming, and environmentally unfriendly technique. SPE is an alternative to address some of these limitations, as it uses significantly less solvent, thereby reducing the environmental impact. Various types of SPE cartridges, mainly polymeric, like Oasis® HLB [21,67], Strata X [69], Oasis MAX [65] and Oasis PRiME HLB [60], as well as C_{18} like Strata C_{18}-E [89] among others, have been employed (see Supplementary Material, **Table S4**).

Nevertheless, the relatively high cost of SPE cartridges limits the widespread adoption of this method [99].

However, there are emerging alternatives to classical SPE that fulfil in a better way the principles of green analytical chemistry, reducing not only costs but also the number of reagents used, experimental steps, and the time employed [104]. Such techniques include SPME, dispersive solid-phase extraction (dSPE), matrix solid-phase dispersion (MSPD), molecularly imprinted polymer solid-phase extraction (MIP-SPE), and MSPE.

SPME is a technique that involves extracting analytes into a fibre coated with a sorbent. This method is well known for its simplicity, speed, and solvent-free nature. In addition, it could be fully automated [105]. In one study, SPME with a polydimethylsiloxane (PDMS) fiber demonstrated its effectiveness in volatile profiling of rare historical preserved foods [60]. BPA was detected and quantified in 29 samples of homemade preserves, at levels ranging from 3.4 to 19.2 $\mu g\ kg^{-1}$. However, it is important to note certain drawbacks of SPME, as for example, its fibres are expensive, fragile, and have a limited lifetime [105]. In addition, sample carryover can be a problem as was found when determining BPs among other EDCs in cheese [99].

Another technique is dSPE, in which an adsorbent is dispersed in the sample instead of being immobilized in a cartridge, allowing for a more dynamic interaction between the adsorbent and analytes. It has some advantages, as it is simple, flexible, and robust, with a reduction in solvent consumption and time, and it does not require repetitive centrifugation, filtration or extraction stages. However, it has the limitation that it cannot be fully automated [105]. dSPE was successfully applied in two works [82,84]. In one of these works [84], an ultrasound assisted extraction (acetone followed by dSPE with primary secondary amine (PSA)) and a GC–MS/MS (HP-5MS, QqQ) analysis was employed for determining BPA, its chlorinated derivatives, and structural analogues in vegetables. The recoveries of eleven BPs ranged from 74 % to 105 %, indicating the effectiveness of dSPE in extracting the analytes from vegetable samples. LOQs were in the range of 0.05 to 1 ng g^{-1}. These low LOQs underscore the high sensitivity of the proposed method, enabling the detection and quantification of BPs at trace levels.

MIP-SPE uses a molecularly imprinted polymer that exhibits selective recognition and interaction with the target compound, prioritising it over other molecules with different properties. This technique presents some advantages, as it is simple, stable, robust, flexible, selective, and resistant to a wide range of pH, solvents, and temperatures. It has also a high extraction efficiency, provides an enhanced sensitivity, and it is a low-cost synthesis approach. However, it also has some disadvantages, as there could be some difficulties with optimisation, the time required for analysis is long, and the potential prevention of long-term use due to analyte build-up [105]. MIP-SPE was successfully applied in some studies [70,73,74,79,86,92], predominantly focusing on the analysis of BPA. Kalogiouri et al. [73], proposed a MIP-SPE–LC–UV (C$_{18}$) method for the extraction of BPA from walnuts. They used a specifically fabricated BPA imprinted sol–gel silica-based hybrid inorganic–organic polymeric sorbent. The analysis revealed BPA concentrations ranging from 0.015 to 0.467 ng g^{-1} in the samples. The results also highlighted the outstanding selectivity of MIP-SPE for BPA when compared to structural analogues such as BPB, BPC, bisphenol E (BPE), bisphenol F (BPF), and BPS.

Another alternative to conventional SPE is MSPD. This technique combines disruption, homogenisation, extraction, and clean-up of the sample in one step, providing advantages such as simplicity and flexibility. In addition, the possibility of emulsion formation is eliminated, solvent consumption is substantially reduced, and it can be used for extracting analytes from both solid and liquid food. The main disadvantage is the lack of automation of the procedure. MSPD was successfully applied for determining three BPs (BPA, BPF, BPS) among other EDCs, including PAEs, in mussels by LC–DAD (PFP-C$_{18}$) [95]. The spiked mussel sample was poured into a glass mortar containing Florisil (dispersing sorbent), Na$_2$SO$_4$ (anhydrous agent), and washed sea sand. It should be also specified that in this study a mixed stationary phase (PFP-C$_{18}$) was used. Fluorinated stationary phases, especially those including a PFP moiety, have become popular alternatives to the more traditional alkyl (C$_8$ and C$_{18}$) phases, and in some case, like the cited study, they are components of a mixed phase because of their orthogonality. The proposed method showed a good performance and provided good recovery percentages (78.5–87.2 %) for the three BPs investigated.

Finally, MSPE, which was employed in two studies [58,86], relies on the use of magnetic adsorbents, which have the advantage of being able to be separated from the solution using an external magnetic field. This property facilitates the isolation of BPs from the sample matrix [106]. In addition, MSPE is simple, fast, cheap, and environmentally friendly with reduced solvent consumption. It also possesses some limitations, as low solubility in water, potential blockage of the active adsorption sites of the sorbent, and the lack of multifunctional coatings [105]. In the study conducted by Xie et al. [58], the analysis of nine BPs in canned beverages, fish, meat samples, and milk powder was performed. They employed MSPE with a novel magnetic sulfonatocalix[6]arene covalent cross-linked porous polymer (Fe$_3$O$_4$@pTMC-SC6A) with core–shell structure, followed by LC–MS/MS (C$_{18}$, QqQ) for determining thirteen kinds of epoxy derivatives, including nine BADGEs. The method demonstrated excellent sensitivity, as evidenced by the low LOQs obtained, which varied from 0.024 to 0.078 ng g^{-1}, as well as satisfactory recoveries, ranging from 74.9 % to 118 %.

Other effective, environmentally friendly, and commonly used sample treatment methods have been employed when determining BPs in food. One of these methods is QuEChERS (quick, easy, cheap, effective, rugged, and safe), which is a widely adopted sample treatment method for determining contaminants in food, due to its simplicity, speed, high sample throughput, the ability to achieve high analyte recoveries for a diverse range of organic chemicals, and the versatility in extracting both polar and non-polar compounds. However, QuEChERS has its own shortcomings. For samples with low water content or high-fat content, the purification effect is not ideal, the extraction efficiency is low, and the purification process has large losses [62]. Several studies have used this technique [62,65,68,70,93,101]. Toptancı et al. [70] used QuEChERs followed by LC–MS/MS (C$_{18}$, QqQ) to analyze seven BPs in fish samples. Acetonitrile was used as extraction solvent in the first stage of the QuEChERS procedure, and magnesium sulfate, PSA and C$_{18}$ were employed in the clean-up of the extracts (dSPE stage). The LOQs ranged from 0.0006 to 0.052 mg kg^{-1}, and recoveries obtained for the studied BPs ranged from 85 % to 105 %. The concentrations of BPs in the fish samples varied, with some being below the LOQ and others ranging up to 0.72 mg kg^{-1}.

DLLME offers advantages such as high enrichment factors, low solvent consumption, and rapid extraction, although it also has certain limitations since each sample can require pH adjustment, filtration, or centrifugation, depending on the sample pre-treatment required, which can increase total sample preparation time [65]. This technique was employed in several studies [59,61,68,75,97–99,102]. In the study conducted by Farajzadeh et al. [98], DLLME followed by GC–FID was employed for determining some migrated chemicals, including BPA, from plastic containers into different drinks and liquids. 2-Propanol was selected as a disperser solvent, while 1,2-dibromoethane and carbon tetrachloride were chosen as extraction solvents. As mentioned earlier, the achieved LOQ was 0.80 $\mu g\ L^{-1}$ and the concentration range of BPA in the analyzed drinks varied between 4.12 and 15.3 $\mu g\ L^{-1}$. However, it is worth noting that the extraction recovery of BPA in this study was relatively low (54 %). Although less commonly used, other sample treatment options include MAE and PLE (both already mentioned in the analysis of MPs), sugaring-out assisted liquid–liquid extraction (SULLE) and an immunoaffinity column (IAC) clean-up.

It should be highlighted that no articles utilizing MAE or PLE as the sole extraction method for BPs analyses were found. MAE was successfully applied in combination with MIP-SPE in a study devoted to determining BPA in canned food by LC–MS (C_{18}, SQ) [79]. The MIP preparation included BPA as template molecule, a mixture of methacrylic acid and 2-vinylpyridine as monomers, ethylene glycol dimethacrylate as cross-linker and acetonitrile as the polymerisation porogen; while a mixture of methanol and water was used to perform the MAE. It is worth noting that in this study, the recovery of BPA was found to be below 60 %. The low recovery rate indicates that alternative extraction methods may be a better approach for the extraction of BPs.

Meanwhile, PLE was employed in a study combined with DLLME for determining EDCs in cheese by LC–DAD (C_{18}) [99]. The analytes were extracted into a water-miscible extraction solvent (acetone) at high pressure and temperature in the presence of carbon tetrachloride as a modifier. After extraction, a volume of the organic phase was removed and dispersed into a sodium chloride solution; in this DLLME step, acetone was the dispersive solvent and carbon tetrachloride the extraction solvent. In this study, the recovery rate of BPA using this combined technique was determined to be 78 %.

SULLE is a phase transition phenomenon first observed in acetonitrile and water mixtures, that acetonitrile can be separated from the mixture with the addition of sugars. This phase separation can be attributed to the replacing of the hydrogen bonding between acetonitrile and water molecules by sugar molecules. Comparing with the traditional phase transition methods, sugaring-out shows advantages of rapid phase separation with more environmentally friendly solvents [88]. However, there are also certain complications that can be encountered when working with SULLE. The formation and management of the sugar phase can introduce additional complexities into the experimental procedure, the optimisation of the SULLE procedure can be challenging (sugar concentration and phase ratio), and there is a risk of contamination of the extraction phase with sugar. A method based on SULLE and HPLC–FLD (C_{18}) has been developed for the determination of BPA and BPB in royal jelly [88]. The optimal SULLE procedure involved extraction with an acetonitrile and water mixture, followed by phase separation with the addition of glucose. LOQs were lower than 45 μg kg^{-1} and average recoveries of BPA and BPB were between 88 % and 98 %. The developed method was applied to the analysis several royal jelly samples, and BPA and BPB were not detected in any of the samples.

Finally, an IAC based on a monoclonal antibody was successfully employed for determining BPA different foodstuffs using UHPLC–MS/MS (C_{18}, QqQ) [66]. The recoveries of BPA from spiked samples ranged from 82.0 % to 104.9 %, with RSDs below 13.8 %. IAC exhibited good reusability, with 40 % column capacity remaining and no significant loss of recovery after 25 application cycles in real sample detection.

In summary, for the determination of BPs in foods, the best option seems to be, according to the existing literature, UHPLC–MS/MS, which shortens the analysis times of conventional LC–MS/MS methods, using reverse-phase mode, with C_{18}-based columns, and mobile phases based on water/salt/acids and methanol or acetonitrile, applied in gradient elution mode. The most suitable detector due to its high sensitivity is the QqQ. GC–MS (non/low polar columns, helium as carrier gas, and temperature programs) can be an alternative, but as it involves derivatisation processes and normally requires longer analysis times, it has therefore been used on considerably fewer occasions. In relation to sample treatments, it is true that although solvent extraction and SPE predominated, the new trend is to use more environmentally-friendly sample treatments when possible, that allow a reduction in costs, stages, and reagents. This is why techniques with DLLME or QuEChERS among others, are emerging as green alternatives to the aforementioned treatments. It is very important to highlight the need to avoid/control as much as possible the contamination of BPs coming from the laboratory material and environment. To do so, very strict cleaning and control measures must be followed, which have been listed previously in this section, and procedural targets must be continuously evaluated.

2.4. Determination of PAEs in food

Several of the studies focusing on the analysis of PAEs in food products conducted between 2018 and 2022 are listed in **Table S5** (see Supplementary Material; [17–19,94,95,97–102,107–143]). GC and LC are the most widely employed techniques for the analysis of PAEs in food samples, since they provide high sensitivity and selectivity. As shown in Fig. 2**A**, the most employed separation technique is GC. This preference stems from the thermal stability and volatile characteristics exhibited by PAEs [144]. The GC analysis of PAEs in food is similar to that of MPs and BPs, with helium as carrier gas, temperature-programmed separations, and non/low-polar stationary phases predominantly employed (HP-5MS [18,96,98,108,125,129]; SLB-5MS [19]; SPB-5MS [75,100]; SE-54 [97,122]; Equity-1 [98,141]; Elite-5MS [101]; TRB-5MS [102]; BP5 and BP-5MS [111,121,138]; CD-5MS [114]; DB-5 and DB-5MS [120,133,134,139]; TG-5MS [128]; ZB-5MS [135]; Optima-5MS [143]. However, it should be noted that intermediate polarity columns have only been used in two works (SLB-35MS [19]; OV-11 [115]; see Fig. 3**B** and **Table S3**, Supplementary Material). Although the use of GC–FID is more common [13,98,111,141] than for MPs and BPs, the specificity and high sensitivity of MS make it the preferred detection technique when analyzing PAEs at low concentration levels. As can be seen in **Table S5** (see Supplementary Material), SQ is the most employed detector in GC analyses [96,98,100,101,108,114,120,122,128,129,133, 135,138,139], while MS/MS (QqQ [18,19,121,125,143]; IT [97,102]) is also popular.

As a special case, the electronic capture detector (ECD) was used for determining PAEs from noodle and rice soup samples [115]. In this work, a novel stainless steel net dumbbell-shaped stir-bar device was successfully developed for the determination of PAEs. This proposed method showed high extraction efficiency (81 % to 110 %), good linearity and sufficiently low LOQs (11 to 32 ng mL^{-1}), while the developed stainless-steel device is affordable, and easy to assemble and operate.

Comprehensive two-dimensional GC (GC × GC)–MS/MS (QqQ), employing a combination of intermediate (SLB-35MS) and non-polar columns (SLB-5MS) [19] was used to analyze nine PAEs in vegetable oil samples without any sample preparation. GC × GC utilizes two different columns with two different stationary phases. In GC × GC, the effluent from the first-dimension column is diverted at regular intervals to the second-dimension column via a modulator [145]. This technique can save time by combining several compound classes into a single analysis, and short-cut sample preparation and clean-up by chromatographically resolving matrix interferences, although it has some limitations as the need to operate the valve continuously at a high temperature, the risk of analyte adsorption or catalytic degradation involving valve materials, and broader peaks [146]. In the mentioned study, the LOQs for the PAEs ranged between 0.06 and 2.10 mg kg^{-1}, and it was found that out of the twenty-seven analyzed oil samples, nine were found to be contaminated with various PAEs, including dipentyl phthalate (DPP) and DEHP.

In relation to the LC analysis of PAEs in food (see Fig. 3**A**), it can be concluded that again the predominant separation mechanism is reverse-phase chromatography with C_{18} columns, and mobile phases composed of water/salts and organic solvents such as methanol or acetonitrile (see Supplementary Material, **Table S5**). Only in some particular cases have other phases been used such as phenyl-hexyl [64], C_8 [112], phenyl [124], or the already mentioned mixed phase C_{18}-PFP [95]. It should be also mentioned that spectrophotometric detectors (UV or DAD; [1,95,99,107,116–118,123,129,137]) have been employed in a greater proportion in relation to the MS detectors than in the case of BPs and MPs. The most common detection wavelength when determining PAEs in food is 225 nm [107,118,129,137], but other wavelengths, such as 210 nm [95], 228 nm [116], 235 nm [99], and 280 nm [123] have been used. For example, LC–DAD (C_{18}) was selected for determining eight PAEs in beverages [107]. This study was the first time a deep eutectic solvent (DES)-based DLLME was used for the extraction of PAEs in these

matrices. Recovery values ranged from 84 to 120 % and the LOQs between 17.2 and 59.4 µg L^{-1}. The use of LC–MS (SQ) [112,124] is much less compared to LC–MS/MS [64,109,110,112,113,119,124,127,130, 132,136,140,142], which could be probably due to the need for determining PAEs at low concentrations. It should be noted that unlike BPs, PAEs are usually analyzed in positive ionisation mode, producing [M + H]$^+$ ions. Within the MS/MS detectors, the majority use of QqQ stands out, while the use of QTRAP [124] and Orbitrap [114,136] was scarce. An example of using an Orbitrap (HRMS) is a study devoted to determining phthalate diesters and their metabolites in seafood species using QuEChERS extraction with UHPLC–HRMS [136]. QuEChERS involved the use of acetonitrile as extracting solvent with salts (magnesium sulfate, sodium chloride, sodium citrate dibasic sesquihydrate, and sodium citrate tribasic dihydrate) followed by a dSPE clean-up with C$_{18}$ and Lipifiltr, which is a push-through purification cartridge specifically developed to remove lipids from fatty samples [136]. Apparent recoveries were up to 79 % for all compounds, matrix effect values ranged from − 8 to − 48 %, and method limits of detection were between 1 and 25 ng g^{-1}. PAEs were found in some of the analyzed samples at concentrations of up to 982 ng g^{-1}.

The analysis of PAEs in any food matrix is a challenge, not only because of the complexity of foods, which may also contain interfering compounds or may produce important matrix effects, but also because PAEs are ubiquitous in any analytical laboratory and, therefore, the analysis of real samples with a low PAE background can be difficult [36]. There are different strategies to try to minimise this problem. For example, the rinsing of plastic materials with organic solvents has been demonstrated to decrease the levels of contamination only slightly. For this reason the use of other materials such as Teflon, aluminium, stainless steel or glassware is recommended. In addition, volumetric glassware should be previously cleaned with oxidising agents, while non-volumetric glassware can be calcined at 450–550 °C, after cleaning with water and organic solvents [18,36,129]. It is also recommended to avoid contamination through the use of high-purity solvents with the lowest levels of contamination and previously purified with aluminium oxide, PAEs-free gloves and pipette tips, the storage of clean materials in a desiccator containing aluminium oxide, regular evaluation of contamination in chromatographic systems, as well as avoiding the use of personal-care products by laboratory staff. Finally and as already mentioned in the case of MPs and BPs, the analysis of procedural blanks during each set of samples evaluated is mandatory, in order to test the contamination coming from the different potential sources [36].

The most employed sample treatments when determining PAEs in food are solvent extraction and SPE, followed by QuEChERS and DLLME (see **Fig. 2B** and **Table S5**, see Supplementary Material). Acetonitrile and methanol are the most used extraction solvents, while classical SPE uses Florisil and PSA [134], and Bond-Elut Plexa (non-polar sorbent [110]) cartridges. However, it is important to note that not only has classical SPE been used in the analysis of PAEs in food products, as in the case of BPs, other related procedures like MSPE, SPME, MIP-SPE, dSPE, and magnetic-dispersive-solid-phase extraction (MdSPE) have also been employed (see **Fig. 2B** and **Table S5**, see Supplementary Material). Because all these techniques have already been discussed in previous sections, further elaboration will not be provided. However, it is worth noting the utilisation of MdSPE. This is an innovative technique that combines the benefits of both dSPE and MSPE, and offers several advantages, including low cost, reduced solvent usage, time efficiency, high enrichment performance, and easy phase separation using a magnet. However, the preparation of the magnetic sorbent requires the synthesis of several steps to create its magnetic propertiest. Also, the presence of magnetic iron oxide in the sorbent network can reduce the selectivity of the extraction method with nonselective extraction of different species in the sample solution, especially in the real samples [146]. MdSPE has been evaluated as an extraction method for determining five PAEs in beverages and plastic bottles by LC–UV (C$_{18}$) [117]. Fe$_3$O$_4$@GC submicrocubes, a novel magnetic carbon material, were successfully designed and constructed by

a facile and time-saving one-step pyrolysis approach using low-cost waste napkins as carbon source, and they were used as MdSPE adsorbents. The proposed method showed good linearity, low LODs (0.01–0.28 µg L^{-1}), and high recoveries (80.0–112.8 %).

Other sample treatments have been employed in previous studies. Two publications reported the use of PLE, although an additional step such as DLLME [99] or SPE [110] was required in both cases. Meanwhile, another article reported the use of magnetic effervescent tablet-based microextraction (MEFT) [116]. This technique offers advantages such as not requiring a physical energy source or dispersive solvent. The dispersion of extractant is based on the release of CO$_2$ generated by the reaction between an acid source and a base source. The rapid and vigorous formation of CO$_2$ bubbles resulting from the effervescent reaction enhances the adsorption and extraction efficiency of analytes. However, the utilisation of this technique is limited, since magnetic effervescent tablets exhibit poor storage and stability characteristics, which are primarily attributed to the reactivity between the acid and alkaline salts contained within them. Consequently, these tablets are prone to moisture absorption and degradation during storage. In the above-mentioned article, MEFTs were used in a MFe$_2$O$_4$-based magnetic ionic liquid effervescent tablet microextraction (MMIL-ETM) for pre-concentration/extraction of PAEs at trace levels in milk matrices that were determined by LC–DAD (C$_{18}$). Under optimised conditions, extraction recoveries for four PAEs (diethyl phthalate (DEP), butyl benzyl phthalate (BBZP), dibutyl phthalate (DBP), bis(2-ethylhexyl) terephthalate (DEHP)) reached as high as 94.8–105.6 %, and LODs were as low as 0.042 µg L^{-1}.

As a conclusion to this section, unlike what happened for the analysis of MPs and BPs, there is no chromatographic technique that clearly predominates over the other. It is true that there are more articles that use GC compared to LC, but the difference is small. In the case of GC, non/low polar columns with helium as carrier gas and temperature program are used, while in the case of LC, reverse-phase mode is used with C$_{18}$ columns, mobile phases typical of this separation mechanism (water/salts/acids and methanol or acetonitrile) and gradient elution mode. Regarding the detection systems, MS (SQ) predominates for GC, and MS/MS (QqQ) for LC. As mentioned previously, the most employed sample treatments when determining PAEs in food are solvent extraction and SPE, followed by QuEChERS and DLLME, although other recently introduced sample treatments that reduced not only the length of the procedure but also the consumption of solvents have been employed (MSPE, SPME, MIP-SPE, dSPE, MdSPE). Finally, it should be remarked that one of the main concerns in the analysis of PAEs is their usual presence in the laboratory. Consequently different strategies (cleaning, use of different materials, calcination, procedural blanks…) should be adopted, in order to minimise the background signals that complicate the analysis of real samples.

2.5. Simultaneous determination of BPs and PAEs in food

It is worth noting that the simultaneous determination of BPs and PAEs in food products is possible, although it has not been commonly reported (see Supplementary Material, **Table S6**), and it has been performed by GC [65,90–98,100–102] and LC [64,94,95,99] (see Fig. 2A). GC–MS (SQ) with non/low polar columns (HP-5MS [96,98]; SE-54 [97]; Equity-1 [98]; SPB-5MS [100]; Elite-5MS [101]; TRB-5MS [102]; see Fig. 3B and **Table S3**, Supplementary Material), helium as carrier gas and temperature programs is the mostly employed choice. By optimising a temperature ramp, it becomes possible to separate both BPs and PAEs effectively. Quantification becomes a straightforward task due to the utilisation of MS with electron impact sources operating at an ionisation energy of 70 eV. This specific ionisation energy allows for the quantification of analytes using their characteristic m/z ions, which can be cross-referenced with libraries. Two of the studies support this information [96,100], as they show that DEP, DBP, BPA, and BBP share identical target ions and have similar qualifier ions.

Furthermore, another study [98] reveals that simultaneous determination of BPA and three PAEs in beverages can be achieved using GC–FID (Equity-1 GC column). The extraction procedure has been already described in *Section 2.2*. However, the authors acknowledge that MS is inherently more sensitive than FID. Thus, while MS is considered the best choice, FID can be a viable alternative when GC–MS is not available.

GC–MS/MS was also employed in two studies [97,102]. In one of these publications [102], the simultaneous determination of six PAEs and BPA in honey samples was achieved by ultrasound vortex-assisted dispersive liquid–liquid microextraction (UVA-DLLME) with benzene followed by GC–MS/MS (TRB-5MS, IT). The LOQs of the method were good enough (<22 ng g^{-1}), and the method was applied to six honey samples. Some PAEs were found in the samples at concentrations below the specific migration limits (SMLs).

In relation to LC analysis, it must be said that reverse phase-mode was used in all cases but with different columns (C$_{18}$ [94,99]; PFP [95]; phenyl-hexyl [64]; see Fig. 3A), and gradient elution mode with conventional mobile-phase components (water/salts and methanol or acetonitrile) was the preferred option. Meanwhile, MS/MS (QTOF [94]; QqQ [64]) and DAD [95,99] were used twice each. According to Tian et al. [94], for the analysis of BPs, electrospray ionisation (ESI) in negative mode is required, while PAEs are to be analyzed in positive mode. However, Sakaki et al. [64] successfully detected and quantified both BPs and PAEs in negative ESI mode. In this study, BPs and PAEs were identified and quantified using UHPLC–MS/MS (phenyl-hexyl, QqQ) in capsule and French press coffee after performing a solvent extraction with ethyl acetate. Low LOQs (0.8 to 2.1 ng mL^{-1}) were obtained for three PAEs (DBP, DEHP, dimethyl terephthalate (DMTP)), indicating the feasibility of performing the entire analysis in negative ion mode. As previously mentioned, the use of LC–DAD is an option to consider to jointly determine BPs and PAEs, as it was possible to use simultaneously several wavelengths. Cañadas et al. [95] conducted a study to determine the optimal wavelength for the analysis of three BPs and three PAEs by using a PFP-C$_{18}$ column after performing an MSPD. They evaluated the analyte areas at different wavelengths (210, 230, 250, 254, and 280 nm), and it was found that the maximum areas for all analytes were obtained at 210 nm, indicating that the analysis could be performed at a single wavelength. However, in a different study in which the compounds were sequentially extracted by using PLE and DLLME [99], different wavelengths were selected for PAEs (235 nm) and BPA (277 nm) after their separation using a C$_{18}$ column. Thus, it can be concluded that the wavelength selection may vary depending on the specific analytes and experimental conditions.

Before commenting on the sample treatments that have been used to simultaneously determine BPs and PAEs, we must remember the need to take extreme precautions in the laboratory to avoid contamination by BPs and PAEs of the samples. Therefore, the strategies previously mentioned in *Sections 2.3 and 2.4* must be applied, which, as can be seen, in many cases are common for both compound families. In relation to the most used sample treatments, they are obviously related to those discussed in previous sections, so it is not surprising that the most used extraction technique was directly related to solvent extraction in any of its varieties [64,94,100] (see Fig. 2B).

Different solvents have been demonstrated to be a good option. Tian et al. [94] were able to determine 11 BPs and 4 PAEs in 168 food composites (fish fillets, chicken breast, canned tuna, leafy vegetables, bread, and butter) with a methodology based on ultrasound-assisted liquid extraction with methanol followed by LC–MS/MS (C$_{18}$, QTOF) determination. Adequate recoveries were obtained in all cases (70–129 %), and it was found that DBP, DEP and DINP were the most frequently detected contaminants in the analysed samples.

Meanwhile, ethyl acetate was selected for extracting three BPs and three PAEs from coffee, and in this case UHPLC–MS/MS (phenyl-hexyl, QqQ) was employed as the determination technique [64]. *n*-Hexane was chosen in a different study devoted to investigating the presence of

plasticizers (PAEs) and BPA in spices and aromatic herbs by GC–MS (SPB-5MS, SQ) [100]. However, a further dSPE (MgSO$_4$ and PSA) was required for cleaning the extracts.

In addition to solvent extraction, DLLME was selected in three studies [97,98,102], in which several PAEs and only one BP (BPA) were determined. In one of these studies [97], UVA-DLLME, using toluene as an extraction solvent, followed by GC–MS/MS (SE-54, IT) was proposed for determining six PAEs and BPA in honey samples. Recoveries ranged from 69 % to 97 %; LOQs were lower than 16 ng g^{-1} in all cases, and residues of six PAEs and BPA were found in some of the samples in a wide concentration range (5–997 ng g^{-1}). It should be mentioned that a similar approach for determining those compounds in honey was proposed by the same authors [102], but in this case benzene was selected as extraction solvent, and a different column (TRB-5MS) was employed. DLLME has proven to be effective for the simultaneous extraction of PAEs and BPA, but there may be a need for further optimisation to improve extraction efficiency for BPA and a broader range of BPs, while ensuring the maintenance of good extraction performance for PAEs. In a different publication [99], PLE combined with DLLME followed by LC–DAD (C$_{18}$) was proposed for determining BPA and three PAEs (DBP, DINP, DEHP) in cheese. Acetone and carbon tetrachloride were the selected solvents for both techniques.

Other techniques employed in the simultaneous analysis of BPs and PAEs in food include MSPD [95], which has been already described in *Section 2.3*, and a modified QuEChERS method [101]. In this latter study, several contaminants, including PAEs and BPA were determined in sunflower oil by GC–MS (Elite-5MS, SQ). Acetonitrile was used for extracting the compounds in combination with magnesium sulfate and sodium chloride, while dSPE was performed with a commercial kit. Bisphenol P (BPP) and DBP residues were found in some the samples.

In summary, the simultaneous determination of BPs and PAEs in food is possible but not commonly reported, which could be due to their different physicochemical properties that influenced the optimal analysis conditions. For this reason, it is not surprising that the conditions reported in the bibliography are a mixture of those that have been found for their separate determination, and that except in some specific applications the number of BPs determined is very low compared to the PAEs. GC (MS or MS/MS) has been mainly selected when the number of PAEs was higher than BPs, while LC (MS/MS or DAD) was more frequently used when BPs predominated. In the case of GC, non/low polar columns with helium as carrier gas and temperature program were used, while in the case of LC, reverse-phase mode was used with C$_{18}$ columns, mobile phases typical of this separation mechanism (water/salts/acids and methanol or acetonitrile) and gradient elution mode. Regarding sample treatments, although the number of articles is the same for solvent extraction and DLLME, the current trend is to use smaller sample volumes and shorter analysis times, making the latter more recommended for simultaneous analysis of PAEs and BPs. However, we must not forget other alternatives related to green chemistry that have been used to a lesser extent such as PLE, MSPD and QuEChERS. Finally, the need for controlling as much as possible the contamination of BPs and PAEs coming from the laboratory material and environment should not be forgotten.

3. Conclusions

A comprehensive summary of analytical methods employed in articles published between 2018 and 2022 for the analysis of MPs, BPs, and PAEs was conducted. The findings of this review indicate a significant interest within the scientific community in studying the presence of MPs and related compounds in food, with a particular emphasis on the use of chromatographic techniques. However, it should be noted that chromatography is less commonly employed for the determination of MPs compared to other spectroscopic techniques. Nevertheless, it remains the main option for the determination of BPs and PAEs. The choice of analytical methods, including sample treatment and determination

techniques, strongly depends on the specific compounds being analysed. For MPs analysis, Py–GC–MS with sample digestion was found to be the preferred technique. LC–MS/MS, accompanied by solvent extraction and classical solid-phase extraction (SPE), was commonly employed for the analysis of BPs. GC–MS was the most frequently used determination technique for PAE analysis, with classical solvent extraction as the preferred sample treatment. It should be also remarked that in the analysis of MPs, BPs and PAEs it is crucial to avoid contamination during all stages of the analytical process, and therefore contamination control measures must be taken to the extreme in the laboratory, while the analysis of procedural blanks is also mandatory.

To sum up, plastic contamination in food is an environmental and public health problem that has gained attention in recent years and can occur both at source and during production and processing, which makes it very difficult to avoid. In view of the data and information summarised in this review article, contamination by these compounds exists in a multitude of foods around the world, and thanks to chromatographic techniques it is possible to identify and quantify them accurately and at low concentrations, which can be of great help in trying to better understand the serious problem of plastic pollution in the food chain. In the not-too-distant future, it is expected that maximum residue or migration limits will be established for many of the compounds mentioned in this review in different foods, since to date there are very few of them, and the use of chromatographic techniques would be essential for their determination. Consequently, all the provided data and information of this review will contribute to expanding knowledge and facilitating the labour of people that would be interested in determining these contaminants in food, ultimately aiming to minimize their negative impact on human health.

Conflict of interest

The authors of this manuscript declare no conflict of interest.

CRediT authorship contribution statement

Beatriz Martín-Gómez: Conceptualization, Investigation, Methodology, Writing – original draft, Writing – review & editing. **J. Stephen Elmore:** Conceptualization, Visualization, Writing – original draft, Writing – review & editing. **Silvia Valverde:** Conceptualization, Visualization, Writing – original draft, Writing – review & editing. **Ana M. Ares:** Conceptualization, Project administration, Supervision, Visualization, Writing – original draft, Writing – review & editing. **José Bernal:** Conceptualization, Investigation, Methodology, Supervision, Visualization, Writing – original draft, Writing – review & editing.

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.

Data availability

Data will be made available on request.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.microc.2024.109903.

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