



Review

# Recent Trends in the Analysis of Chemical Contaminants in Beverages

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**Abstract:** Chemical contaminants should not be present in beverages for human consumption, but could eventually be ingested by consumers as they may appear naturally from the environment or be produced by anthropogenic sources. These contaminants could belong to many different chemical sources, including heavy metals, amines, bisphenols, phthalates, pesticides, perfluorinated compounds, inks, ethyl carbamate, and others. It is well known that these hazardous chemicals in beverages can represent a severe threat by the potential risk of generating diseases to humans if no strict quality control is applied during beverages processing. This review compiles the most updated knowledge of the presence of potential contaminants in various types of beverages (both alcoholic and non-alcoholic), as well as in their containers, to prevent undesired migration. Special attention is given to the extraction and pre-concentration techniques applied to these samples, as well as to the analytical techniques necessary for the determination of chemicals with a potential contaminant effect. Finally, an overview of the current legislation is carried out, as well as future trends of research in this field.

**Keywords:** chemical contaminants; residues; beverages; food contact materials; food additives; analytical methods

## 1. Introduction

By definition, beverages are potable liquids for human consumption, which are essential for many physiological functions, such as giving energy and refreshing the body [1]. In general terms, “beverages” include many different products, such as milk, coffee, tea, juices, and alcoholic products, which humans commonly consume in their daily lives [2–7]. In agreement with recent literature [8–10], the most frequent drinks currently used in the human diet can be classified into two categories: Alcoholic and non-alcoholic beverages. A brief classification of beverages is shown in Figure 1.

The vast majority of beverages, including alcoholic and non-alcoholic drinks, are based on large amounts of water. Nonetheless, water is not classified as a beverage, although in this review, bottled mineral water will be treated as a special kind of beverage, as suggested by some authors [1].

The alcoholic group includes beverages, both distilled and non-distilled, mainly obtained from fruits or grains, with wine and beer having high rates of consumption worldwide, being typical examples of fruit-produced and cereal-produced beverages, respectively [11–13].



**Figure 1.** Classification of beverages taking into account some features such as source, alcohol, or CO<sub>2</sub> content. Adapted from [8].

On the other hand, non-alcoholic beverages are subdivided into carbonated and non-carbonated drinks, including vegetable and fruit juices, prepared or embedded water-based beverages, such as coffee and tea, and dairy products. Carbonated soft drinks are those to which carbon dioxide has been added intentionally to produce a special kind of non-alcoholic liquor, including sodas, cola, lemonade, lemon/lime, tonic, etc.

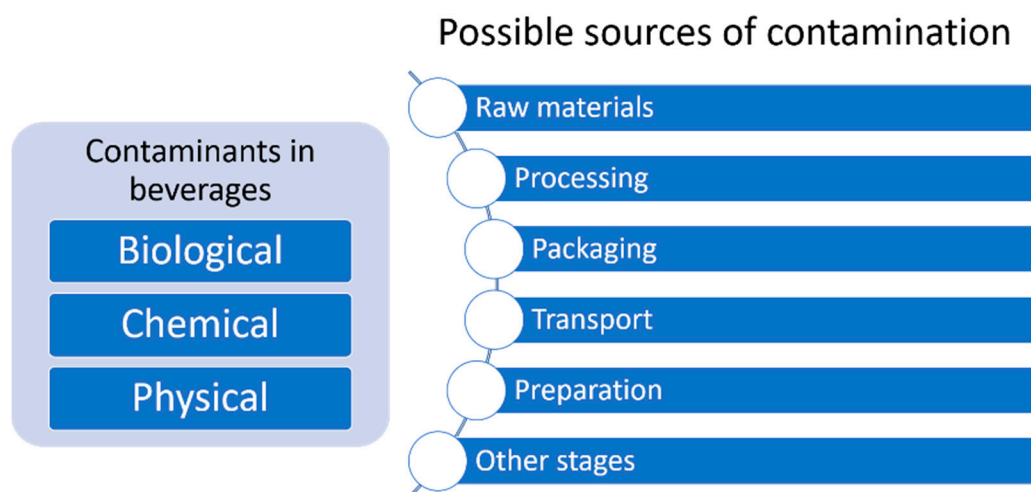
It is well-known that the beverages market is global, including all categories already mentioned, either alcoholic or non-alcoholic. This industry is in continuous search of innovative and safer products to provide consumers with high-quality products and new tastes that are absolutely free of any potentially hazardous chemicals. In particular, regulatory compliance with the quality of beverages is an important objective to be achieved, not only for companies but also for authorities, policy-makers, and, of course, scientists, as the proposal of new products is linked to scientific advances in food and beverages science and technology. The quality control of beverages is strictly necessary to guarantee their safety and consumers health, as well as to improve their nutritional value [14,15].

The term “contaminants” in beverages includes those compounds that are dangerous to health, whether chemical, physical, or microorganisms. Potential contamination is one of the main issues for consumers when selecting beverages for their use, as it could represent a risk for their health and well-being [16]. Although chemical and biological contaminants are strictly banned in beverages formulation, they can sometimes be unintentionally formed during processing, packaging, and distribution processes. In addition, physical contaminants such as foreign bodies that may be present in beverages must be taken into account (glass, wood, metal fragments, etc.). These may come from raw materials, but also from unintentional contamination from personnel or devices used during the processing and packing of the final product [14].

Particularly, biological contaminants, such as bacteria, fungi, protozoa, and viruses, can enter directly into human cells, triggering severe infectious diseases. Beverages with a strong nutritional characteristic stand out as potential sources for the growth and persistence of this type of contaminant [17]. Therefore, it is essential to avoid their presence in beverages while their early identification is necessary to avoid their spread through the human body after ingestion [10].

Humans may also be exposed to various hazardous chemical pollutants (which are discussed in detail in the following section). The acute toxicity caused by this type of contaminant is forcing

food and drinks companies to perform strict quality controls to their products before putting them into the market [14]. However, exposure to low levels of chemical contaminants for prolonged periods is still a clear risk for human health. Many of these substances have carcinogenic, mutagenic, and teratogenic potential [18], and exposure can cause chronic diseases, representing a serious risk to human health [10,19]. A brief classification of contaminants in beverages and their possible sources are shown in Figure 2.



**Figure 2.** Brief classification of contaminants in beverages and their possible sources.

Food packaging facilitates the storage, handling, transport, and distribution of beverages. These concepts are related to inert materials acting as “passive” barriers to separate the packaged product from the environment. Similarly, it must be considered that packaging materials should prevent unintentional migration of harmful chemicals to food and beverages [20]. However, the increasing use of new packaging materials, most of them with specific functionalities, could represent a risk by the potential release of unknown chemicals to food and beverages [21]. These chemical contaminants are directly dependent on the material nature [22] and can be associated with every possible material in beverages packaging [20].

Polymers are mostly used in beverage packaging, with higher rates than other materials, such as glass and tin, mainly due to their low cost and versatility. In general terms, plastic packaging consists of not only individual polymers but also polymeric multilayers where different materials are joined to increase their performance as packaging materials. Polypropylene (PP), high (HDPE)- and low-density polyethylene (LDPE), poly (ethylene terephthalate) (PET), poly (vinyl chloride) (PVC), and polystyrene (PS) are some of the most usual commodities used in beverages packaging [21]. Several contaminants, such as heavy metals, polymer additives, and degradation by-products, can be considered potential contaminants in beverages [23–25].

Cans and other similar containers are the main metal-based packaging materials for beverages, particularly aluminum and steel cans. As most aluminum cans are coated with polymer-based coatings in their inner part, direct contact between the beverage and the metal container is quite infrequent. However, it should be highlighted that migration from the coating could take place. Nevertheless, metals-based packaging materials should be studied to guarantee food and beverages safety by avoiding manganese, chromium, tin, zinc, lead, or cadmium migration [26].

This review focuses on discussing the main contaminants potentially present in beverages and their containers, as well as their possible detection techniques, emphasizing discussion of the most recent studies in this area. The effects on human health of these chemicals will also be discussed, including contaminants and their metabolites, which are also an important part of the risk associated with their presence in beverages [27].

## 2. Chemical Contaminants

The presence of undesired chemicals in beverages and their effect on human health have been largely studied in the last few years. Some of them could be present in the raw materials before processing and end up in the final product. However, the most frequent way for incorporation of chemicals into beverages is through chemical reactions involving some components of the drink itself or through release from packaging materials. Phthalates and heavy metals are described as two of the main contaminants in beverages, as recently reported [18,19]. This section focuses on some of the most important groups of potential chemical contaminants in beverages. They could be organized into two families: (i) Organic compounds (such as phthalates, bisphenol A, and related compounds, primary aromatic amines, perfluorinated compounds, UV ink photoinitiators, ethyl carbamate, and the recently described non-intentionally added substances (NIAS)); and (ii) inorganic compounds (mostly heavy metals). All those chemicals and their main effects are summarized below along with the extraction techniques and analytical methods necessary for their identification and quantification. Their determination requires faster, efficient, reliable, economical, and environmentally friendly analytical methods, which scientists are currently developing to respond to the urgent demand for safe beverages without any trace of contaminants in their composition. The use of innovative instrumentation and protocols is necessary to improve the most relevant analytical figures of merit, such as precision, sensitivity, and specificity, among others [28].

### 2.1. Inorganic Contaminants

#### Heavy Metals

Heavy metals are elements naturally found in different environmental matrices in small concentrations (generally lower than  $1 \text{ mg kg}^{-1}$ ), while some of them play important roles for living organisms [29]. However, some heavy metal ions are quite toxic and are considered the foremost inorganic contaminants with high potential to cause negative effects on human health if recommended levels are exceeded. Therefore, they should be monitored frequently in food and beverages to verify their safety for human consumption [16].

Raw materials and added sugars are the most common source of heavy metals in beverages, but their presence as traces in by-products of agrochemical treatments, such as pesticides and fungicides, packaging materials, or as the result of processing technologies should also be considered [21,25].

Arsenic (As), cadmium (Cd), chromium (Cr), lead (Pb), and mercury (Hg) can be considered the most toxic elements potentially present in beverages even at very low concentrations. These elements could induce mutagenicity and carcinogenicity among other negative effects [29]. On the other hand, cobalt (Co), calcium (Ca), copper (Cu), magnesium (Mg), iron (Fe), manganese (Mn), molybdenum (Mo), potassium (K), nickel (Ni), selenium (Se), zinc (Zn), and tin (Sn) are essential elements in many important biological processes within the human body [16]. However, even these elements at high concentrations can cause severe damage to human health [29]. In recent years, the determination of other metals, such as platinum (Pt), rhodium (Rh), and vanadium (V), with potential negative effects on human health, has raised some interest [19]. Pb, Cd, Hg, and Sn have been considered contaminants by the European Commission (EC) regulations [30], while the World Health Organization (WHO) listed, in 2010, Hg, As, Cd, and Pb in its list of the main chemicals with high public health concerns [31]. Their identification and quantification were carried out through different analytical methods. Prior to detection of the analytes, procedures for sample preparation or extraction should be followed to prepare samples for analysis. The main goal of selecting specific analytical methods for heavy metals determination is to obtain the best results fast, with no contamination, and using the minimum amounts of solvents and reagents [16].

Considering that each element has different emission and absorption spectra, atomic spectrometry techniques are the best selection for their identification and quantification. Inductively coupled plasma (ICP) and flame atomic absorption spectrometry (FAAS) are widely used techniques for such purpose.

The electrothermal atomic absorption spectrometry (AAS) technique, as well as graphite furnace atomic absorption spectrometry (GFAAS), are currently increasing their importance in these determinations with improved detection and quantification limits with respect to FAAS [29].

A pre-concentration stage prior to the determination by atomic spectroscopic techniques is often necessary in the analysis of beverage samples to increase the analytes concentration while improving the detection limit, precision, and sensitivity by eliminating matrix interferences. Different pre-concentration methods for heavy metals have been reported. Some of them are co-precipitation, cloud point extraction (CPE), ion exchange, liquid–liquid extraction (LLE), solid phase extraction (SPE), dispersive liquid–liquid microextraction (DLLME), or ultrasound-assisted ionic liquid dispersive liquid–liquid microextraction (UA/IL/DLLME), among others [29].

The main reports for the determination of heavy metals in beverages are shown in Table 1, including the combination of extraction and pre-treatment processes with various analytical techniques. The combination of CPE with FAAS was used by Gürkan et al. [32] and Altunay et al. [33] for the determination of Sn and Sb species in canned beverages, either alcoholic or non-alcoholic, and in PET bottles, respectively. These authors reported detection limits of 0.33, 1.68, and 4.28  $\mu\text{g L}^{-1}$  for Sn (IV), Sb, (III), and Sb (V), respectively. Similarly, CPE has been used for the formation of ion mating complexes and determination of As in water and alcoholic and non-alcoholic beverages, using UV-Vis with 1.14  $\mu\text{g L}^{-1}$  as the detection limit for As (V) [34]. Matiazzi et al. [35] reported the determination of Sb in juice flavors bottled in PET through AAS, while Uluozlu et al. [36] used co-precipitation as the most adequate pre-treatment for Sn analysis in mineral water and fruit juices (detection limit 0.013  $\mu\text{g L}^{-1}$ ). The innovative application of solid surface fluorescence (SSF) to determine Sb and Zn was proposed by Talio et al. [37,38]. Authors reported limits of detection and quantification of  $0.36 \times 10^{-3}$  and  $1.29 \times 10^{-3}$   $\mu\text{g L}^{-1}$  in Zn (II) and 0.08 and 0.24  $\mu\text{g L}^{-1}$  in Sb (III), respectively.

Inductively coupled plasma combined with either mass spectrometry (ICP/MS) or optical emission spectrometry (ICP/OES) has increased their use in the determination of metals due to their many advantages, particularly possibilities to determine many elements in a single run and their excellent detection limits [16]. However, the use of ICP-based techniques shows certain drawbacks, such as their high operating and maintenance costs, as well as high consumption of argon [39].

Different sample treatments for the determination of metals, such as Mn, Fe, Cu, Pb, Zn, Cd, As, Sn, Al, and Hg, in beverages by using ICP/MS and ICP/OES have been proposed. Acid digestion is the most used preparation strategy for beverages either directly or assisted by microwaves (MW), as reported by Görür et al. [40] and Fathabad et al. [41]. Dry ashes (DA) formation was proposed by Abdhel-Rahman et al. [25] to quantify Fe, Mn, Cd, Pb, Ni, Cr, and Cu by ICP/OES in milk derivatives, carbonated drinks, and juices, either bottled in plastic or in cans. Finally, Biata et al. [42] developed a simple and fast UA/IL/DLLME method for the pre-concentration of Sn and Sb traces in beverages with further quantification by ICP/OES.

On the other hand, Whitt et al. [43,44] evaluated different packaging materials based on recycled PET (rPET) with ICP/OES to determine their content in Cd, Cr, Ni, Sb, and Pb. Similarly, Dutra et al. [45] used ICP/MS to quantify the heavy metals content in rPET, recycled HDPE (rHDPE), and rHDPE-multi-layer systems, obtaining values below the legislation limits for the first two materials, while in the rHDPE multi-layer, a higher concentration was observed, which was attributed to the manufacturing process. Similarly, Mertoglu-Elmas et al. [46] determined Pb, Cd, Zn, Ni, and Cu in various materials used in the production of corrugated cardboard by ICP/MS.

On the other hand, a DLLME method for the extraction and pre-concentration of Cd and Pb from soft drinks with further quantification by GFAAS was developed by Mandlate et al. [47]. Limits of detection (LOD) were 0.006 and 0.072  $\text{ng L}^{-1}$  for Cd and Pb, respectively. Finally, a method based on high-resolution continuum source flame atomic absorption spectrometry (HR-CS/FAAS) was developed by Fernández-López et al. [48] to analyze Zn and Cu in a large variety of beverages with excellent results. LODs were 0.040 and 0.016  $\text{mg L}^{-1}$  for Zn and Cu, respectively.

**Table 1.** Inorganic contaminants in beverages and analytical techniques used for their determination.

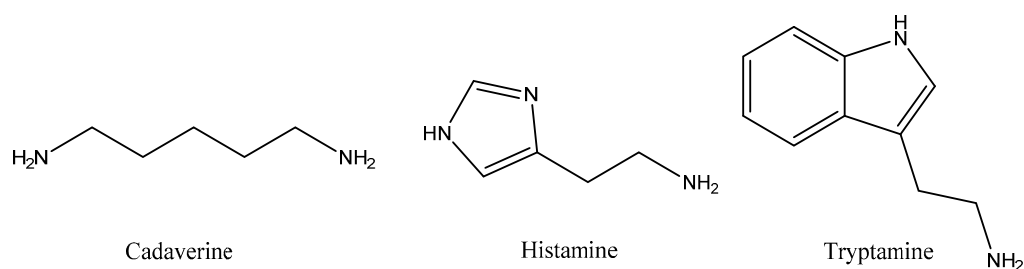
Chemical Contaminant	Packaging Material	Beverage	Sample Treatment/	Analytical Method	Ref.
Fe, Mn, Cd, Pb, Ni, Cr, Cu	Plastics, cans	Carbonated drinks, flavored yogurt, juice	DA	ICP/OES	[25]
Sn	Cans	Non-alcoholic, alcoholic beverages	CPE	FAAS	[32]
As	-	Drinking water, soft drinks, alcoholic beverages	CPE	UV-Vis	[34]
Fe, Mn, Zn, Cu, Pb	-	Black and green tea	Acid digestion	ICP/OES	[40]
Sb	PET	Mineral water and juice flavors	-	HR/CS/AAS	[35]
Sb, Zn	PET	Mineral water, flavored water and beverages	SPE	SSF	[23,24]
Sb, Sn	PET, cans	Beverages	UA/IL/DLLME	ICP/OES	[42]
Cd, Hg, Sn, Al, Pb, As	Glass	Fruit juices	MW/acid digestion	ICP/OES	[41]
Sn	-	Mineral water and fruit juices	Co-precipitation	GFAAS	[36]
Sb	PET	Non-alcoholic, alcoholic beverages	CPE	FAAS	[33]
Cd, Cr, Ni, Sb, Pb	rPET	-	MW-acid digestion	ICP/OES	[28,29]
As, Al, Ba, Cr, Fe, Mn, Mo, Ni, Pb, Se	rPET, rHDPE, rHDPE multi-layer	-	-	ICP/MS	[45]
Pb, Cd, Zn, Ni, Cu	rBoard	-	Acid digestion	ICP/OES	[46]
Cd, Pb	-	Soft drinks	DLLME	GFAAS	[47]
Zn, Cu	-	Alcoholic and non-alcoholic beverages	-	HR/CS/FAAS	[48]

## 2.2. Organic Contaminants in Beverages

### 2.2.1. Amines

Biogenic amines (BAs) are basic compounds that are synthesized through some metabolic routes. They are minor constituents of non-fermented raw materials for beverages at low concentrations. Nevertheless, large quantities of these amines have been occasionally detected in specific cases in both beverages and fermented food as a consequence of the presence of contaminating microflora. Therefore, BAs have been traditionally employed as indicators of the sanitary quality of beverages by their microbiota alteration. The chemical structure of the main BAs can be aliphatic, aromatic, or heterocyclic [49] (Figure 3).





**Figure 3.** Chemical structure of some biogenic amines (Bas).

Low quantities of BAs are crucial for many human vital processes. However, their ingestion at high concentrations can cause major illnesses through triggering alterations in some important physiological functions, such as cell development, brain activity, corporal temperature, and digestive pH regulation and the immunological response to bacteria and viruses [50]. By consequence, the presence of high concentrations of BAs could lead to headaches, lung distresses, heart palpitation, vomiting, blood pressure disturbances, anaphylaxis, and, lastly, death [51]. Amines with vasoactive activity, such as  $\beta$ -phenylalanine (Phe), tryptamine (Try), and tyramine (Tyr), alter the cardiovascular system, whereas amines with psychoactive function, such as histamine (His), cadaverine (Cad), and putrescine (Put), have their influence on the nervous system. Tyr and His, the most toxic BAs, can be found in beverages, so companies and consumers should pay special attention to them [51]. The effects of BAs on humans are not only dependent on their intrinsic properties, type, and amount, but also on the age, genus, and previous diseases of each individual.

BAs are usually found in beverages, and ripeness or fermentation can contribute to some increase in their concentration. This is particularly critical in wines and fruit nectars, where Cad, spermidine (Spr), spermine (Spm), and Put have been found [52]. No restrictions have been imposed for BAs in beverages, although some European countries have set up recommendations for His in wine not exceeding 2–10 mg L<sup>-1</sup> [49].

Primary aromatic amines (PAAs), such as aniline, 2,6-diaminotoluene, 4,4'-methylenedianiline, 2,4-diaminotoluene, or 1,5-diaminonaphthalene, can be found in beverages. These molecules can migrate from packaging materials and are considered toxic compounds and suspected human carcinogens [53]. These compounds can be found in water and food simulants, as well as in beverages that have been in contact with kitchen utensils or plastic laminates/flexible multilayer food packaging materials [54,55].

The choice of analytical techniques for determination of BAs in beverages should take into account the presence of structurally similar compounds that could interfere even at low concentrations. For this reason, SPE and LLE have been considered adequate pre-treatment systems prior to BAs analysis. More recently, micro and miniaturized extraction methods have been reported as useful and eco-friendly analytical tools for BAs extraction. Enzyme-linked immunosorbent (ELISA) methods constitute a rapid and easier alternative for such purpose [56,57].

Due to the fact that amines are not chromophores and their volatility is quite low, a derivatization step is needed [40–42]. It has been reported that these previous steps in the analytical procedure improve resolution in liquid chromatography methods [58–61].

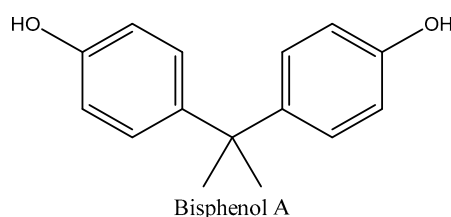
Several methods have been reported for amines detection in beverages (Table 2). In most of them, high-performance liquid chromatography (HPLC), gas chromatography (GC), and capillary electrophoresis (CE) are used [62]. CE methods are rapid and efficient with low reagents consumption. For example, the capillary electrophoresis mass spectrometry method (CE/MS/MS) was applied for the simultaneous assessment of BAs in beer and wine [62]. Emerging electrochemical techniques with different surface/modified electrodes are taking their place as alternatives for amines analysis by their easy procedures and short times required for analysis. A potentiometric sensor with molecularly printed nanoparticles formed through solid-phase printing has also been proposed [63], while electrochemical biosensors are being used for BAs determinations, many of them based on amperometric detection [64].

**Table 2.** Amines in beverages and analytical procedures used for their determination.

Chemical Contaminant	Beverage	Pre-Treatment/ Derivatization	Analytical Method	Ref.
Put, Cad, Try, Phe, Spr, Spm, His, Tyr and 1,3-diaminopropane	Grape wine. Rice wine Beer	LLE	HPLC/DAD	[57]
His, Tyr, Try, Phe	White and red wines	Precolumn. 1-fluoro-2-nitro-4-(trifluoromethyl) benzene	HPLC/DAD	[60]
Put, His, Try, Cad, Agm, Spr, Spm, Phe	Wine. Beer	Precolumn Tosyl chloride	HPLC/MS/MS	[65]
Cad, Try, Tyr, His, Spm	Wine. Beer	Precolumn Fluorenylmethyl	HPLC/DAD	[56]
Tyr, Put, Try, Phe, Spm, Cysteamine, Methylamine	Fruit juices Beer	Precolumn 1-naphthylisothiocyanate	HPLC/DAD	[66]
Put, Cad, Spm, Tyr, Phe, His	Wines	-	HPLC/FLD	[66]
Put, Spm, His, Tyr, Cad, Spm, Phe, Serotonin	Coffee	Precolumn. Dansyl chloride	HPLC/DAD	[67]
Tyr, Phe, Put, Cad, His, Tyr, Spr, Spm	-	-	UHPLC/MS/MS	[68]
22 PAAs	Food simulant	Trifluoroacetic anhydride	HPLC/MS/MS	[54]
8 PAAs	-	Trifluoroacetic anhydride derivatization	GC/MS	[54]

### 2.2.2. Bisphenols

Bisphenols consist of two linked hydroxyphenyl groups with hydroxyl functionalities. Bisphenol A (BPA) is the most important compound under this category by many advantageous properties, particularly the antioxidant characteristic (Figure 4). BPA could act as a catalyzer of polymerization reactions, particularly polycarbonates and epoxy resins. In addition, it is extensively employed in the production of phenol resins, polyacrylates, and polyesters. BPA and bisphenol F (BPF) are not only used as precursors of epoxy resins, but also as PVC additives in organosol resins, as they are used to get rid of the spare hydrochloric acid produced in reactions involving PVC [69].

**Figure 4.** Chemical structure of bisphenol A (BPA).

BPA could be found in beverages, making necessary its determination, as the Food and Agricultural Organization (FAO) and the WHO reported negative effects of BPA on animals in concentrations lower than 5 mg kg<sup>-1</sup> [70]. The restriction of migration of BPA in food was fixed at 0.6 µg g<sup>-1</sup> by the EC [71], and, furthermore, the 2011/8/EU Directive banned the use of BPA in polycarbonate-based children beverage containers [72]. The EC also banned the use of bisphenol F diglycidyl ether (BFDGE) in 2005 [73], while no tolerable limits were set for bisphenol B (BPB). In 2015, the European Food Safety Authority (EFSA) issued a new evaluation of BPA exposure to humans, reducing the tolerable daily intake (TDI) of BPA from 50 to 4 µg kg<sup>-1</sup> body mass/day.



Some restrictions in the use of BPA in packaging materials forced companies to replace it by safer chemicals in their formulations and products. For instance, products labeled as “BPA-free” are becoming frequent in canned beverages by containing alternative chemicals, with properties like those of BPA but reduced toxicity and lower estrogenic activity. Among them, BPF, bisphenol S (BPS), and BPB are those with a higher presence as BPA substitutes. These compounds can be found in both soft and canned drinks. However, their use should also be controlled as they show endocrine functions and toxicological profiles similar to BPA, showing some carcinogenic effects.

BPA has been found in infant food, soft drinks [74], and energy drinks [69,75], and it is particularly present in resins employed as inner coatings in metal cans to avoid corrosion. This extensive use could result in beverage contamination due to the presence of metals or bacteria. BPA, which penetrates food and beverages from these epoxy resins, has also been found in recycled paper, probably due to the use of printing dyes.

Electrochemical methods have been reported as those most sensitive and reliable for the BPA detection, as they are fast, repeatable, reproducible, cheap, and do not need pre-treatment steps prior to analysis [1]. For example, BPA was detected in modified polyglycerol esters (PGE) from teething and feeding bottles for babies [76]. Surface analysis was carried out by scanning electron microscopy (SEM) and analysis was performed by electrochemical methods. These methods showed a low LOD of  $134 \text{ pg L}^{-1}$ . In addition, a carbon electrode with nanoparticles obtained by reduction from cuprous oxide-wrapped graphene oxide ( $\text{Cu}_2\text{O-rGO}$ ) was designed as an electrochemical BPA sensor with a very low LOD,  $5.3 \times 10^{-8} \text{ M}$  [77].

Another electrochemical sensor for detecting BPA in water with differential pulse voltammetry (DPV) has been recently developed [78]. This sensor was fabricated with multi-walled carbon nanotubes (MWCNT) and gold nanoparticles (AuNP) forming a coating for a glass-carbon electrode. Good recoveries were obtained with this sensor. A NiO/CNT nanocomposite/ionic liquid carbon paste electrode (NiO/CNT/IL/CPE) was also developed for BPA quantification [78]. Authors compared the unmodified and modified electrodes and reported that the LOD obtained was  $0.04 \text{ }\mu\text{M}$ . Table 3 summarizes examples of electrochemical sensors for BPA found in the literature.

A new specific, reliable, and highly sensitive method for determination of several bisphenols in canned energy drinks was described by Gallo et al. [69]. Authors used ultra-performance liquid chromatography (UPLC) with fluorescence detection (FLD), after pre-treatment by using molecularly imprinted polymers (MIPs), allowing the simultaneous determination of BPA, BPF, BFDGE, BPB, and bisphenol A diglycidyl ether (BADGE) down to  $0.50 \text{ ng mL}^{-1}$ . This method was applied to the determination of these contaminants in forty energy drinks. MIP are specific chromatographic solid stationary phases with active sites, creating a 3D system with high specificity and even selectivity for the molecule to be trapped. This method also allowed purification of the obtained product, making this method more effective than SPE procedures [69]. Other authors reported the existence of matrix effects in the determination of bisphenols due to the co-elution of other species, which can interfere. These authors proposed several strategies to solve these problems, either improving the sample-treatment procedures or increasing the resolution of the chromatographic separation by changing the gradient program [79].

BPA and perfluorinated compounds were extracted from children beverages and were enriched using solid phase microextraction (SPME). The SPME device was covered by a very thin film of polyaniline and nanocomposites based on multi-walled carbon nanotubes (PANI/MWCNTs) by means of electrochemical deposition. Analytes were desorbed and ionized inside a glass capillary by electrospray ionization (ESI) and ions were, furthermore, analyzed by tandem mass spectrometry [80].

A SPE method combining cationic exchange and reversed-phase mechanisms was enhanced to offer a selective extraction and purification of BPA and analogs. Pyridine-3-sulfonyl chloride can be used as the most adequate derivatizing agent for bisphenols, in particular by their ionization efficiency in ESI [81].

**Table 3.** Bisphenols in beverages and analytical procedures for their determination.

Chemical Contaminant	Packaging Material	Beverage	Sample Treatment/Extraction Process	Analytical Method	Ref.
BPA	Nursing bottle	-	-	Amperometry Electrode: Tyr/Au@PDA-RGO-Chit/GCE LOD = 0.10 nM	[82]
BPA	-	Milk	-	DPV Electrode: Na-doped WO <sub>3</sub> /CPE LOD = 0.028 μM	[83]
BPA	Baby bottles Drinking bottles	-	-	FIA Electrode: MWCNTs-OH/GCE LOD = 0.18 μM	[84]
BPA	Milk bag Plastic juice box	-	-	DPV Electrode: ILs@HPS-Ni/CdFe <sub>2</sub> O <sub>4</sub> /GCE LOD: 4.55 nM	[85]
BPA	-	Milk	-	EIS Electrode: MCH/Aptamers/Au-NPs/BDD LOD = 1 fM	[86]
BPA	Plastic bottles	Water samples	-	FIA Electrode: SPCE/PEDOT/BMIMBr LOD: 0.02 μM	[87]
BPA	PC drinking bottle	-	-	LSV Electrode: CMK-3/nano-CILPE LOD: 0.05 μM	[88]
BPA	Water bottle	-	-	SWV Electrode: NiO/CNT/IL/CPE LOD: 0.04 μM	[89]
BPA	-	Water	-	DPV Electrode: ILs-LDH/GCE LOD: 4.6 nM	[90]

Table 3. Cont.

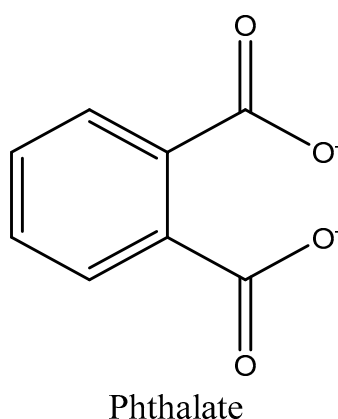
Chemical Contaminant	Packaging Material	Beverage	Sample Treatment/Extraction Process	Analytical Method	Ref.
BPA	PC drinking bottles		-	LSV Electrode: MWNTs/Au/paper electrode LOD: 0.13 $\mu$ M	[88]
BPA	-	Milk	-	DPV Electrode: MWCNTs-PEI/GCE LOD: 3.3 nM	[91]
BPA	-	Milk	-	DPV Electrode: rGO-Fc-NH <sub>2</sub> /AuNPs/GCE LOD: 2 nM	[92]
BPA	-	Milk carton samples	-	DPV Electrode: GR/Au-Tyr-CS/GCE LOD: 1 nM	[93]
BPA	-	Soda Milk	-	LSV Electrode: Gr-IL/GCE LOD: 8.0 nM	[94]
BPA	Plastic feeding bottles	-	-	CV Transducer: TYR/TiO <sub>2</sub> /MWCNTs/PDDA LOD: 1 $\mu$ M	[95]
BPA	Mineral water bottle	-	-	DPV Transducer: $\beta$ -CD/ILCPE LOD: 4.16 nM	[95]
BPA	Polycarbonate drinking bottle	-	-	CV Transducer: AuNPs/MoS <sub>2</sub> /GCE LOD: 0.005 $\mu$ M	[96]
BPA	Plastic bottled drinking water	Canned beverages	-	Derivative voltammetry Transducer: MIP-GR/ABPE LOD: 6.0 nM	[97]

Table 3. Cont.

Chemical Contaminant	Packaging Material	Beverage	Sample Treatment/Extraction Process	Analytical Method	Ref.
BPA	Water bottle		-	SVW Transducer: ZnO/CNTs/IL/CPE LOD: 9.0 nM	[98]
BPA	-	Milk Mineralized water samples	-	DPV Transducer: ERGO-MN202/CS/GCE LOD: 1.02 nM	[83]
BPA	-	Milk	-	CV Transducer: GNPs/GR/GCE LOD: 5 nM	[83]
BPA	Drink bottle	-	-	CV Transducer: CTAB/CPE LOD: 0.1 $\mu$ M	[99]
BPA	-	Mineral water	-	CV Transducer: f-SWCNT/PC4/GCE LOD: 0.032 $\mu$ M	[100]
BPA	-	Milk	-	DPV Transducer: Bi2WO6-CPE LOD: 20 nM	[101]
5 bisphenols	-	40 canned energy drinks	MIPs	UPLC/FLD LOQ: Down to 0.50 ng mL <sup>-1</sup>	[69]
8 bisphenols	-	Soft drinks	-	HPLC/FLD LOD: 5.52–21.37 ng mL <sup>-1</sup>	[75]
BPA	-	Infant drinks	SPME	Ion/MS/MS	[80]
12 bisphenols	-	Alcoholic and non-alcoholic beverages	Mixed-mode solid-phase extraction and stable-isotope dilution	HPLC/MS/MS LOD: 1.6–27.9 ng L <sup>-1</sup>	[81]
BPA	Cardboard box Plastic canned Plastic Cardboard box with plastic cap and metal	Milk Soft drinks Bottle water Juice	LLE with acetonitrile, n-hexane, isopropanol, acetone:heptane mixtures	UPLC/MS/MS	[102]

### 2.2.3. Phthalates

Phthalates are diesters of 1,2-benzene dicarboxylic acid (or ortho-phthalic acid) and they are organic lipophilic compounds with the structure shown in Figure 5. Phthalates are mainly used as plasticizers in the formulation of polyethylene, PVC, and other rigid polymeric materials to increase their flexibility by their molecular interaction with polymeric chains [103], also increasing their durability [104]. As phthalates are not linked with polymeric chains, they are easily released from the matrix and, consequently, the possibilities of contamination to beverages should be studied [105].



**Figure 5.** Chemical structure of phthalates.

A common characteristic of phthalates is that they can be quickly hydrolyzed to monoesters, which can also be transformed into their metabolites. It has been reported that phthalates analysis in urine can be used as a biomarker to assess human exposure to these compounds [70]. It should also be considered that the amount of phthalates in spirits and wine is limited according to the EU Regulation No 10/2011 of 14 January 2011 on plastic materials [106] with strict specific migration limits of 0.3, 1.5, and 30 mg L<sup>-1</sup> for di(butyl phthalate) (DBP), di(ethyl hexyl phthalate) (DEHP), and butyl benzyl phthalate (BBP), respectively.

Therefore, innovative analytical techniques and methods have been recently reported for the detection of phthalates in beverages, as shown in Table 4. Most reports for monitoring phthalates focus on simple samples, e.g., phthalates migration into water from plastic bottles. Nevertheless, the migration of phthalates from polymers-based packages to complex beverages matrices, such as juice, carbonated beverages, milk, etc., is difficult to evaluate, as their release during the usual times of processing and use in beverages is small enough to remain at values below the LOD of the selected techniques [103]. The most common analytical methods used for phthalates determination involve GC coupled to mass spectrometry detection (GC/MS), which permit the quantification of phthalates in beverages at concentrations as low as µg kg<sup>-1</sup>. This is the reason why these methodologies usually include a pre-concentration step, for example, LLE or SPE, as well as micro-extraction methods, such as SPME, liquid-phase microextraction (LPME), or DLLME [103,107].

A previous set of pre-treatment operations including cutting into small pieces is necessary for the determination of phthalates released from packaging materials. A further extraction step with n-hexane/acetone (1:1; v:v) with centrifugation and further recovery of analytes from the supernatant has also been reported prior to injection into GC/MS [105].

**Table 4.** Phthalates in beverages and analytical methods for their determination.

Chemical Contaminant	Packaging Material	Beverage	Analytical Method	Ref.
Dimethyl phthalate Diethyl phthalate	Plastics, paperboard	Hydroalcoholic beverages	GC/FID GC/MS	[105]
Diisopropyl phthalate	Plastics, paperboard	Soybean milk Wine	SPE/MIP US/LLME/GC	[103,104]
Dibutyl phthalate	Paper	Bottled milk	GC/MS	[103,108]
Diisobutyl phthalate	Paper, plastics, paperboard	Soybean milk Hydroalcoholic beverages	SPE/MIP GC/FID GC/MS	[108]
Benzylbutyl phthalate	Plastic	Bottled milk Wine	US/LLME/GC	[103,105,108]
Dipentyl phthalate	Plastics, cardboard	Hydroalcoholic beverages	GC	[108]
Dicyclohexyl phthalate	Plastics, cardboard	Wine	GC	[108]
Dihexyl phthalate	paper	Wine	GC	[108]
Diocetyl phthalate, bis(2-ethylhexyl) phthalate	Paper, plastics, cardboard	Bottled milk	GC	[108]
PHAs		Wine coffee	GC/MS	[19]
6 phthalates		Bottled milk	GC/FID LOD: 0.64–0.79 ng g <sup>-1</sup>	[102]
8 phthalates		Wines	HPLC/MS/MS LOQ: 1.6–9.8 µg L <sup>-1</sup>	[104]
3 phthalates	Polypropylene Baby Bottles	Milk simulants	FTIR GC/MS	[109]
5 phthalates	-	Soybean milk	GC/MS	[110]
8 phthalates	Tetra brick®	Beer, soft drinks, juices, soup, milk	GC/EI/MS GC/MS	[105]
11 phthalates	-	Bottled beverages	SFC/UV LOD = 1.5–3.0 ng mL <sup>-1</sup>	[111]



#### 2.2.4. Pesticides

Chemical pesticides are increasingly employed in agricultural practices. However, their use can lead to environmental problems and the appearance of unexpected contaminants in the final products. In general terms, these pesticide residues include insecticides, herbicides, and fungicides based on organophosphorus, organochlorine, pyrethrin, pyrethroids, carbendazim, carbamates, carbofurans, and acetamiprid. There is great concern about their extensive use and the possibilities to be present in beverages, as some of their raw materials are vegetal products. For instance, wine producers use a large amount of pesticides in vineyards to increase production, with the risk of allowing some residues to reach the final product if they are not well removed during processing. Fruit juice and beer producers have similar problems with pesticides. Organizations such as FAO and WHO established a joint Commission to coordinate food standards, including beverages, and they established some universal maximum levels for pesticides expressed in  $\text{mg kg}^{-1}$ . [112].

Identification and quantification of pesticide residues in beverage matrices can be accomplished by applying different pre-treatment and pre-concentration methods to permit a clear reduction in the complexity of sample treatments while increasing the accuracy of the analysis.

The proposal of new sustainable extraction techniques where the use of solvents is greatly reduced has resulted in a wide range of novel and alternative procedures based on LPME, using hollow fiber membranes (HF/LPME), SPE and its miniaturization through SPME, dispersive solid-phase extraction (DSPE), DLLME, matrix solid-phase dispersion (MSPD), and single-drop microextraction (SDME). Chromatographic techniques are the most usual selection for separation of individual compounds from a complex matrix, such as those from beverages, as they provide excellent results in terms of separation, resolution, and selectivity [112]. GC/MS, tandem GC/MS/MS, HPLC/MS, and tandem HPLC/MS/MS are the most common techniques for such determinations, as shown in Table 5. Cladière et al. [113] selected a total of 32 compounds, including pesticides, and used LLE with HPLC with time-of-flight MS (HPLC/MS/ToF) for their determination, obtaining LODs between 0.1 and 6.9  $\text{ng mL}^{-1}$ , at the best and worst case scenarios, respectively. Bolaños et al. [114] also used HF/LPME hyphenated with HPLC/MS/MS for the determination of 51 pesticides from alcohol beverages obtaining low values of LOD in the interval between 0.01 and 5.61  $\mu\text{g L}^{-1}$ . Dias et al. [115] evaluated the pesticides content in red wine, obtaining low limits of quantification (LOQ) (1–50  $\mu\text{g kg}^{-1}$ ) by using HPLC/MS/MS without any pre-treatment. Pérez-Ortega et al. [116] included a preliminary pre-concentration stage by SPE before determining 60 pesticides in wine with HPLC/MS/ToF. They obtained excellent results with low LOD values (0.04–3.80  $\mu\text{g L}^{-1}$ ). Ferrer et al. [117] reported the determination of 53 pesticides in fruit juices by direct injection to HPLC/MS/MS with LOQs obtained of the order of  $\mu\text{g L}^{-1}$ . Durak et al. [118] evaluated the presence of five pesticides in tea samples by switchable solvent liquid phase microextraction followed by gas chromatography with mass spectrometry detection (SS/LPME/GC/MS), obtaining LOD values between 0.44 and 1.5  $\text{ng mL}^{-1}$ . Anjos et al. [119] developed an efficient procedure for the analysis of pesticides in samples of coconut juice, using SDME in combination with GC/MS. This procedure obtained good LODs (0.1 and 0.88  $\mu\text{g L}^{-1}$ ) and LOQs (1.21 and 6.69  $\mu\text{g L}^{-1}$ ). Mohebbi et al. [120] introduced the combination of DSPE and DLLME for the analysis of pesticides in juices at trace levels ( $\text{ng mL}^{-1}$ ) before its quantification by GC/FID. They obtained good results with low LODs (0.32–0.76  $\text{ng mL}^{-1}$ ) and LOQs between 1.1 and 2.6  $\text{ng mL}^{-1}$ . A rapid and sensitive method was developed by Zhu et al. [99] to determine 131 pesticides in tea samples using GC/MS/MS preceded by DSPE with LOD values in the 0.5–5  $\mu\text{g kg}^{-1}$  range. Huang et al. [121] also used a novel modified DSPE protocol with HPLC/MS/MS detection for the determination of 102 pesticides in green tea with LOD and LOQ values between 0.03 and 15  $\mu\text{g kg}^{-1}$  and 0.1 and 50  $\mu\text{g kg}^{-1}$ , respectively. Similarly, DSPE was used by Lozano et al. for the analysis of 86 pesticides in tea by HPLC/MS/MS and GC/MS/MS [122]. Authors highlighted the necessity to develop selective extraction methods to ensure good recoveries and help to decrease matrix effects. Omote et al. [123] selected a total of 277 pesticides to be determined in beer at the 0.001–0.02  $\text{mg kg}^{-1}$  level. They used LLE and subsequent solid–liquid extraction (SLE), prior to quantification by HPLC/MS/MS. Dušek et al. [124] carried out the evaluation of the

persistence of 58 pesticides in beer, which were extracted using the Quick, Easy, Cheap, Effective, Rugged, and Safe method called QuEChERS for subsequent identification and quantification by HPLC/MS/MS. They determined that thermostable pesticides were the most potential risk category due to their persistence in the final product and consequent human exposure. Hengel et al. [125] evaluated the presence of 72 pesticides in various beer samples, known to be previously used in the commercial production of hops. They obtained good results with low LODs (0.024–0.322 ng mL<sup>-1</sup>) and a LOQ of 0.5 ng mL<sup>-1</sup>. The method used was LLE followed by detection using HPLC/ESI/MS/MS. Nagatomi et al. [126] developed an SPE-based method, followed by HPLC/MS/MS, to simultaneously analyze five pesticides on 15 different canned beer samples, obtaining high sensitivity with a LOQ of 10 mg kg<sup>-1</sup>. Inoue et al. [127] also evaluated the persistence of more than 300 pesticides throughout the beer production process and their persistence in the final product. The method consisted of an extraction using QuEChERS with subsequent identification and quantification by HPLC/MS/MS. On the other hand, the combination of SPE with HPLC/MS/ToF was used by Gilbert-López et al. [128,129] for the determination of a large group of representative pesticides in fruit-based soft drinks. The obtained LOQs varied in the µg L<sup>-1</sup> concentration level.

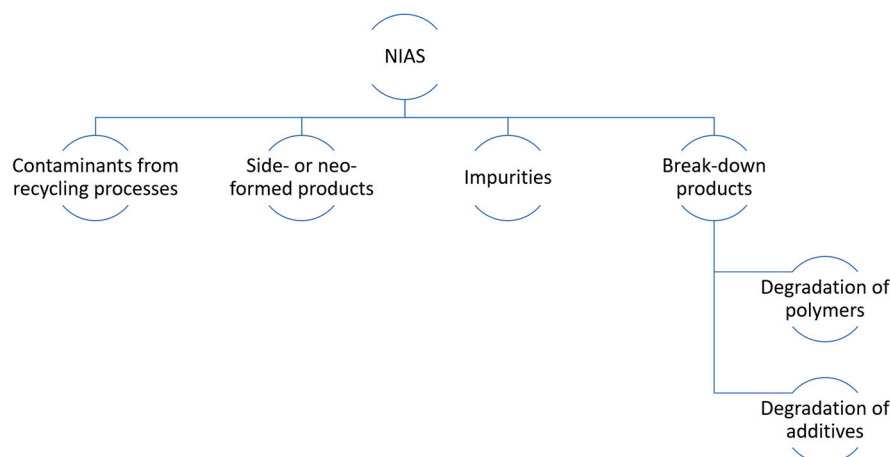
**Table 5.** Pesticides in beverages and analytical techniques for their determination.

Chemical Contaminant	Beverage	Sample Treatment/Extraction Process	Analytical Method	Ref.
21 pesticides	Tea	LLE	UHPLC/MS/ToF	[113]
21 pesticides	Tea	HF/LPME	UHPLC/MS/MS	[114]
185 pesticides	Red wine	-	UHPLC/MS/MS	[115]
60 pesticides	Wine	SPE	HPLC/MS/ToF	[116]
53 pesticides	Fruit juices	-	HPLC/MS/MS	[117]
5 pesticides	Tea	SS/LPME	GC/MS	[118]
19 pesticides	Coconut water	SDME	GC/MS	[119]
9 pesticides	Fruit juices	DSPE DLLME	GC/FID	[120]
131 pesticides	Tea	DSPE	GC/MS/MS	[99]
102 pesticides	Tea	DSPE	HPLC/MS/MS	[121]
86 pesticides	Tea	DSPE	HPLC/MS/MS GC/MS/MS	[122]
277 pesticides	Beer	LLE, SLE	HPLC/MS/MS	[123]
58 pesticides	Beer	QuEChERS	HPLC/MS/MS	[124]
72 pesticides	Beer	LLE	HPLC/MS/MS	[125]
5 pesticides	Beer, Barley Tea	SPE	HPLC/MS/MS	[126]
300 pesticides	Beer	QuEChERS	HPLC/MS/MS	[127]
30 pesticides	Fruit-based soft drinks	SPE	HPLC/MS/ToF	[128]
33 pesticides	Fruit-based soft drinks	SPE	HPLC/MS/ToF	[129]

### 2.2.5. Non-Intentionally Added Substances (NIAS)

NIAS are substances that appear in food and beverages packaging materials, but they have not been purposely added during processing. NIAS can be formed in specific processing and use conditions, and they can be released from the material's surface in contact with food and beverages (Figure 6). They could arise as a consequence of reactions between different components in packaging materials and their own degradation, fundamentally due to impurities present in the precursor materials [130].

Many NIAS are detected by using high-sensitivity analytical techniques, but the elucidation of their chemical structure is often a challenge. It is expected that a greater number of these chemicals will be detected in the coming years, while the development of new analytical methods continues to respond to these unknown compounds [131].



**Figure 6.** General classification of non-intentionally added substances (NIAS) that can be found in beverages; adapted from [132].

The EU Regulation 10/2011 on materials in contact with food [106] establishes how NIAS can be formed. This was the first time that NIAS was defined and subject to a risk assessment study and legislation. However, no levels of migration or exposure to NIAS are established in these regulations. Threshold limits have been set up to some undesired substances in contact with beverages that should not migrate in levels above  $10 \mu\text{g kg}^{-1}$  of food [133]. Standard protocols for NIAS identification have not been proposed, and just some methods to carry out direct analysis on food-preserving material or food/beverages simulants have been reported. Depending on the target analyte, gas or liquid chromatography can be employed, generally coupled to MS to allow the accurate assignment of NIAS chemical structures. Nevertheless, a large number of these compounds have not been identified yet and much work is still necessary in this area. However, despite these drawbacks, identification of NIAS is a trending topic in materials research, as well as a complete risk assessment of these compounds. In general terms, the focus is currently on NIAS with a molecular weight lower than 1000 Da as it is considered that compounds with a higher molecular weight have not the necessary characteristics to effectively reach food and beverages by migration [131].

Several analytical approaches have been recently proposed for the determination of NIAS in plastics in contact with beverages (Table 6). Methods based on chromatographic techniques, such as HPLC/MS, GC/MS, or GC/MS with headspace solid phase microextraction (HSSPME), have been proposed [26]. However, other methods related to direct analysis, such as an atmospheric solid analysis probe, direct analysis in real time, or desorption ionization, are currently under development [131]. Spectroscopy techniques, like Raman, have been proposed for the identification of NIAS, as no sample pre-treatment is required [134].

Ramos et al. [130] evaluated the potential of HPLC with a quadrupole time-of-flight-triple quadrupole mass spectrometer detector (HPLC/Q/TOF/MS/MS) for the identification of 26 potential migrants in different packaging materials, such as LDPE with nylon composites and other PE-based materials. Many compounds identified by them were previously named as NIAS, although just one of them, i.e., caprolactam, appeared in the EU-Regulation No 10/2011 [106]. They also stated that PE-based materials released NIAS at concentrations higher than those detailed in the legislation. Athenstädt et al. [135] identified the migration of NIAS from polyurethane (PU) by using GC/MS, as well as HPLC/MS with ESI. Thirteen different chemicals were detected as NIAS, of which 11 were identified as cyclic esters. Felix et al. [136] used headspace/SPME and GC/MS to identify potential

NIAS migrating from PU adhesives joined to PET, polyamide, PP, PE, and PE/vinyl ethyl alcohol films. In this case, two food simulants were used and more than 63 potential NIAS were detected. Caprolactam was also identified in the same way by Ramos et al. [130]. The use of UHPLC/MS/TOF was reported by Canellas et al. [137] to study migrating compounds from sealants in food cans whose concentration was above the legislative values.

Bauer et al. identified 42 migrants, including 39 NIAS, by HPLC/Q/TOF/MS [138]. Martínez-Bueno et al. [139] proposed a multi-analytical approach to evaluate previously undetected NIAS that could release from a monolayer film with polylactic acid (PLA), polylimonene (PL), and zinc oxide nanoparticles (ZnO NPs) by ICP/MS to identify the possible transfer of zinc oxide nanoparticles from the polymeric matrix as an antimicrobial agent. They reported the detection of seven NIAS, three by GC/MS and four by HPLC/MS. In another study conducted by Osorio et al. [140], 21 NIAS, including three oligomers, were identified as potential migrants by GC/MS. Garcia-Ibarra et al. [141] analyzed 12 plastic materials in contact with beverages to evaluate their possible migration to simulants by using GC/MS. Around a hundred different organic compounds were detected, including NIAS, most of them not included in the current legislation. Migration of various compounds from almost 30 PP films used in beverages packaging was analyzed by UPLC/MS/QTOF by Vera et al. [142]. In addition, 76 compounds were identified and 75% of them were considered NIAS produced as a result of the degradation of the raw materials used in the polymer formulations. In addition, Portesi et al. [134] used rapid and non-destructive confocal Raman spectroscopy for semi-quantitative analysis of NIAS in LDPE. Four major NIAS were identified, and these results were confirmed by ICP/MS analysis. Finally, Cincotta et al. [143] developed a method based on HS/SPME/GC/MS to determine the release of NIAS based on the time of storage in PET bottles. They obtained good results in terms of linearity, precision, and detection with low LOD (0.05–0.17  $\mu\text{g L}^{-1}$ ).

**Table 6.** NIAS in beverages and the analytical method used for their determination.

Chemical Contaminant	Packaging Material	Beverage	Sample Treatment/Extraction Process	Analytical Method	Ref.
26 potential migrants	LDPE+ nylon, PE based material	-	Migration test	HPLC/Q/TOF/MS/MS	[130]
13 potential migrants	PU	-	Migration test	GC/MS HPLC/MS	[135]
63 potential migrants	PU and films of different plastics	-	Migration test	HSSPME/GC/MS	[136]
7 potential migrants	Soda can sealers	-	Migration test	UHPLC/IMS/QTOF/MS	[137]
42 potential migrants	Multilayer packaging PET/Al/PE	-	Migration test	HPLC/Q/TOF/MS	[138]
7 potential migrants	Monolayer film PLA/PL/ZnO NPs	-	Migration test	GC/MS HPLC/MS	[139]
7 potential migrants	Biopolymers based on starch and PLA	-	Migration test	GC/MS	[140]
100 potential migrants	Different plastic films	-	Migration test	GC/MS	[141]
76 potential migrants	PP films	-	Migration test	UHPLC/Q/TOF/MS	[142]
17 potential migrants	LDPE	-	-	Raman spectroscopy	[134]
26 potential migrants	PET bottles	-	-	HSSPME/GC/MS	[143]

### 2.2.6. Other Contaminants in Beverages

Perfluorinated compounds (PFCs) are another type of chemical contaminant in beverages. These compounds in general, particularly the two main compounds under this class, i.e., perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS), are used in multiple applications, but they show the serious shortcoming of being persistent in the environment [144]. Direct exposure of beverages to PFOS and PFOA could occur due to processing and use [79], although with their precursors, such as fluorotelomeric alcohols (FTOH), perfluorooctane sulfonamides (FOSA), and perfluorooctane sulfonamidoethanols (FOSE). It should be noted that polyfluoroalkyl phosphate (PAP) esters are FTOH precursors and could thus lead to PFOA. These compounds have been the object of some attention by their extensive use in containers made of cardboard and paper, such as milk and fruit juice commercial multilayers [54]. These compounds have a carbon–fluoride bond, which is chemically stable and gives them a high possibility of persistence in the environment, which could result in bioaccumulation in biological tissues. It has also been described that PFOS and PFOA can negatively influence the endocrine system, as well as acting as potential tumor promoters [145]. Therefore, EFSA has established a TDI value of 150 and 1500 ng kg<sup>-1</sup> body weight/day for PFOS and PFOA, respectively [146].

Guerranti et al. [144] reported the accurate determination of PFOS and PFOA in milk by using HPLC/ESI/MS with concentrations in real samples lower than LOD. Haug et al. [147] also determined PFCs in 21 food and beverages samples from Norway. About 20 PFCs were detected, including PFOA and PFOS. Previous extraction was performed by alkaline digestion and liquid extraction, followed by SPE and further detection by HPLC/ESI/MS. Zabaleta et al. [145] proposed the use of high-performance liquid chromatography with a triple quadrupole mass spectrometer (HPLC/QqQ/MS/MS) to determine multiple PFCs and potential precursors in packaging materials for milk and coffee. Similarly, the concentrations of various PFCs in almost 400 food samples (including beverages) were determined with HPLC/MS/MS and different pre-treatment methods by Heo et al. [61]. The concentration was variable up to maximum values of 48.3 ng g<sup>-1</sup>, corresponding to PFOA in beverages.

Inks are traditionally applied to food packaging materials including those used in beverages. They usually comprise polymeric resins, pigments, dyes, and other compounds. The main goal of printing inks is to provide technical data about the packaged food in the label or the external part of the package. The use of these compounds is regulated by the European Printing Ink Association, where hundreds of compounds are recommended for their use in this application [148]. It is known that the migration of these components from the container to beverages can occur during processing or storage [54]. It should also be noted that the use of UV-cured printing inks has increased compared to solvent-based inks, which have traditionally been considered dangerous for the environment and for humans [149].

Aznar et al. reported the presence of 17 migrants from inks normally used in beverages packaging by using UPLC/Q/TOF/MS [148]. Blanco-Zubiaguirre et al. [150] evaluated the migration of 97 chemicals, including photoinitiators, from paper and cardboard materials in food simulants by HPLC/QqQ and high-performance liquid chromatography Orbitrap (HPLC/qOrbitrap), obtaining significant concentrations of benzophenone and 4-phenylbenzophenone in values close to limits set by the EU legislation. Chang et al. developed a HPLC/MS/MS procedure to identify 30 different photoinitiators in ten types of packaging materials for fruit juice [151]. The sample was treated with QuEChERS and some compounds were quantified at concentrations between 2.2 and 152.9 ng g<sup>-1</sup>. A QuEChERS/LC/MS/MS method was also used by Gallart-Ayala et al. for the determination of 11 inks in packaged foods (including fruit juices, water, and wine) at ng kg<sup>-1</sup> levels [152]. In addition, 13 compounds were identified after migration in PE-based materials in different food simulants by Zhang et al. [153] who used SFC/PDA/MS/MS. They obtained low LOD values, between 0.02 and 2.16 µg L<sup>-1</sup>. Finally, Vavrous et al. [154] studied the detection of around 70 potential contaminants (including photoinitiators) in materials in contact with food and beverages, using ultrasonic extraction by QuEChERS and HPLC/MS/MS with low LOQ (between 0.0013 and 0.22 mg kg<sup>-1</sup>).



Urethane or ethyl carbamate (EC) is the ethyl ester of carbamic acid and it is sometimes present as a by-product in beverages processing in which processes like fermentation, distillation, and long-term storage are included. EC can be introduced into the human body through fermented beverages, including spirits, for instance, beers, wines, and brandy, where it is considered a potential carcinogenic agent. However, EC is present not only in alcoholic beverages, but also in non-alcoholics, like fruit juice or milk [155,156]. EC is soluble in water, ethanol, and many other organic solvents, making it usual in medical practices, particularly as an antineoplastic agent [156]. EC can be present in distilled alcoholic beverages at concentration levels between 0.01 and 12 mg L<sup>-1</sup> depending on their source [157]. The main EC precursors are basically urea and other similar compounds, such as citrulline, cyanogen, carbamyl phosphate, and diethyl pyrocarbonate. Urea thermal degradation results in cyanic acid, which further reacts with ethanol to form EC. It is also known that large amounts of nitrogen fertilizers in crops contribute to increase the amount of EC precursors.

LLE, SPE, SPME, and HSSPME are powerful extraction tools that allow pre-concentration of EC from beverages. Dichloromethane was the most widely used extraction solvent in LLE. Recently, advanced ultrasound-assisted emulsification microextraction (USAEME) has been applied for EC extraction and pre-concentration. Effective microextraction by packed sorbent (MEPS) has been applied for the determination of EC in fortified wines [158]. GC coupled with several detection options, e.g., flame ionization detection (FID), alkali flame ionization detection (AFID), hall electrolytic conductivity detection (HECD), and MS, has been reported as the most used analytical technique for EC determination. Fourier-transform infrared (FTIR) spectroscopy is another technique developed to determine EC in combination with partial least-squares (PLS) chemometric methods. This strategy provides additional information on the amount of hydrocyanic acid, providing an estimation of the total quantities of EC formed when stored. Nevertheless, as a result of its lack of accuracy, FTIR is just suggested for semi-quantitative rather than quantitative determination of EC. UHPLC/MS/MS methods have also been proposed by their very simple sample preparation and the use of non-organic solvents [156]. HPLC/FLD with a previous derivatization step was applied to the EC detection in several alcoholic beverages, such as beer, wine, sake, soju, whisky, or brandy [85]. Finally, a robust method for rapid extraction and accurate quantification of EC in wine and brandy based on the incorporation of MIP and surface-enhanced Raman spectroscopy (MIPs/SERS) was reported [159].

### 3. Conclusions and Future Trends

Recent developments on analysis approaches for the major groups of chemical contaminants in beverages and their packaging materials have been reviewed. This type of chemical hazard belongs to many groups (organic and inorganic), including heavy metals, amines, bisphenols, phthalates, pesticides, perfluorinated compounds, inks, ethyl carbamate, and non-intentionally added substances. Regardless of the pre-treatment operations, GC/MS is mostly employed for the analysis of phthalates, BPA, and NIAS in beverages and packaging materials, while HPLC/MS/MS is being used as the most adequate technique for more polar molecules (e.g., PFCs, PAAS, and photoinitiators).

Nevertheless, analytical challenges should still be faced by researchers in this field, such as the development of more robust and suitable techniques to offer results with higher analytical quality by increasing accuracy, sensitivity, specificity, and analysis time. Microextraction methods are considered promising in sample preparation, as they help to improve the analytical figures of merit for the determination of contaminants in beverages, most of them highly volatile chemicals, while decreasing LODs and LOQs.

Successful implementation of these methods could provide multiple advantages associated with miniaturization and green analytical chemistry. Simplification of procedures, as well as reduction of analysis times and wastes generated in the laboratory, are still challenging issues in these determinations. The proposal of analytical methodologies that are environmentally friendly is pursued in these developments to finally propose methodologies in the control of contaminants in beverages by simple, sensitive, portable, robust, and reliable instrumental techniques.



Matrix effects in the study of contaminants in beverages are another key issue, and it is necessary to focus on this research to solve this problem due to the complex nature and composition of many beverages. Consequently, researchers must take into account the suitability of analytical methods to each matrix and try to continue improving both sample pre-treatments and extraction procedures in order to pre-concentrate analytes and limit interferences caused by other matrix compounds.

However, continuous research on packaged beverages is essential to ensure that no traces of contaminants could be present in their formulations. These studies could be a challenge for researchers due to the presence of NIAS, as the importance of these pollutants has increased the awareness in the scientific community, making necessary the proposal of new and robust methods to detect this new group of contaminants in beverages.

Needless to say, scientists should review the current legislation, comparing with analytical innovations regarding contaminants in beverages and packaging materials. Therefore, introduction of proactive measures that assess the reliable and fast determination of these pollutants is a must in the current state of research in this area. However, this work will not take effect if there is no support from society, private, and political institutions, for the detection and characterization of these kind of compounds, while establishing action agreements to establish the necessary measures to control beverages contaminants coming from packaging materials.

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