Polycyclic Aromatic Hydrocarbons (PAHs) Sample Preparation and Analysis in Beverages: A Review

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Abstract

The monitoring of food contaminants is of interests to both food regulatory bodies and the consumers. This literature review covers polycyclic aromatic hydrocarbons (PAHs) with regard to their background, sources of exposures, and occurrence in food and environment as well as health hazards. Furthermore, analytical methods focusing on the analysis of PAHs in tea, coffee, milk, and alcoholic samples for the last 16 years are presented. Numerous experimental methods have been developed aiming to obtain better limits of detections (LODs) and percent recoveries as well as to reduce solvent consumption and laborious work. These include information such as the selected PAHs analyzed, food matrix of PAHs, methods of extraction, cleanup procedure, LOD, limits of quantitation (LOQ), and percent recovery. For the analysis of tea, coffee, milk, and alcoholic samples, a majority of the research papers focused on the 16 US Environmental Protection Agency PAHs, while PAH4, PAH8, and methylated PAHs were also of interests. Extraction methods range from the classic Soxhlet extraction and liquid–liquid extraction to newer methods such as QuEChERS, dispersive solid-phase microextraction, and magnetic solid-phase extraction. The cleanup methods involved mainly the use of column chromatography and SPE filled with either silica or Florisil adsorbents. Gas chromatography and liquid chromatography coupled with mass spectrometry or fluorescence detectors are the main analytical instruments used. A majority of the selected combined methods used are able to achieve LODs and percent recoveries in the ranges of 0.01–5 ug/kg and 70–110%, respectively, for the analysis of tea, coffee, milk, and alcoholic samples.

Keywords Polycyclic aromatic hydrocarbons · Tea · Coffee · Milk · Alcohols · GC–MS

Introduction

Food contaminants such as pesticide residues, mycotoxins, and environmental pollutants can be commonly found in raw materials for feed and food production processes due to non-identical worldwide weather, harvesting, and storage conditions (Nielen and Marvin 2008). In addition, food preparation steps such as cooking at a high temperature will potentially release food processing contaminants such

as polycyclic aromatic hydrocarbons (PAHs), acrylamide, and heterocyclic amines into the food (Rey-Salgueiro et al. 2009). This has led to an increasing interest in the monitoring of these contaminants in food, which can be quite challenging due to the complexity of food matrixes in food products.

In this review, one of the food processing contaminants, namely PAHs, will be the main focus. The ever increasing in the number of articles regarding the presence of PAHs in foodstuffs has garnered attention worldwide due to its ubiquity, and the resulting health hazards that can arise through ingestion, inhalation, and dermal contact with PAHs. This review summarizes the studies carried out by researchers who utilized different extraction methods to determine the presence of PAHs in beverages. In addition, researchers are also focusing on developing simpler and alternative extraction, cleanup, and analytical methods, which yield good recoveries and limits of detection (LODs).

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Background

PAHs are highly hydrophobic and organic lipophilic compounds with fused aromatic rings mainly composed of hydrogen and carbon atoms (Essumang et al. 2013). These compounds are semi- or non-volatile in nature, ubiquitous, and known for their carcinogenic and mutagenic potential (Ledesma et al. 2014). They range from two to up to ten fused aromatic rings (Huang and Penning 2014). Molecular sizes of PAHs determine their vapor pressures and therefore affecting their dispersal of in the environment. Heavy PAHs (four or more fused rings) have lower volatility and adsorb on combustion particles like soot whereas light PAHs (less than four fused rings) are extremely volatile compounds, existing mostly in their gaseous state and widely distributed (Lammel et al. 2010).

Sander and Wise (2020) stated that 660 PAHs were revealed according to the NIST Special Publication 922 “Polycyclic Aromatic Hydrocarbon Structure Index.” Nevertheless, 16 PAHs such as naphthalene (NPH), acenaphthylene (ACY), acenaphthene (ACP), fluorene (FLR), phenanthrene (PHE), anthracene (ANT), fluoranthene (FA), pyrene (PY), chrysene (CHR), benz(a)anthracene (BaA), benz(b)fluoranthene (BbFA), benzo(k)fluoranthene (BkFA), benz(a)pyrene (BaP), indeno(1,2,3-cd)pyrene (IP), benzo(g,h,i)perylene (BghiP), and dibenzo(a,h)anthracene (DBahA) were being chosen as priority pollutants by the United States Environmental Protection Agency (USEPA) (Tamakawa 2004). The 16 USEPA PAHs are mostly hydrophobic and non-polar solids with high melting and boiling points.

The Formation Mechanism of PAHs

PAHs are known to be formed by condensation processes of smaller organic compounds in absence of oxygen by either pyrolysis or pyrosynthesis (Ciemniak et al. 2019). Pyrolysis (pyro meaning “fire” and lysis for “separating”) is the decomposition of organic material at elevated temperatures in an inert atmosphere and it involves irreversible change of chemical composition and physical phase (Viegas et al. 2019). At elevated temperatures above 200 °C, pyrolysis will occur and organic compounds will be fragmented, hence producing free radicals which unite to form stable polycyclic aromatic compounds (pyrosynthesis). The amount of PAHs formed is directly proportional with an increase in temperature, which affects both the structure and diversity of PAHs formation (Chen and Chen 2001).

Toxicological Aspects and Human Health Effects

The major health concern that arises from the ingestion of food contaminated with PAHs is the increased in risks of cancer (Huang and Penning 2014). Nevertheless, it was found that some PAHs are not carcinogenic on their own but may function as co-carcinogens (do not usually cause cancer on their own but promote the activity of other carcinogens in causing cancer). From the research experiments carried out on animals, the common areas of cancer formation after the consumption of PAHs are located within the digestive system (Huang and Penning 2014). However, PAHs tend to exist in complex mixtures of varying compositions, and hence the evaluation of their associated health risks can be challenging.

The Agency for Toxic Substances and Disease Registry (ATSDR 2009a) shared that besides contributing to the carcinogenicity and mutagenicity of the PAHs in experimented animals, certain PAHs can produce many other detrimental health effects, including reproductive, developmental, immunotoxic, and neurologic effects. However, the carcinogenic, mutagenic, and teratogenic effects of PAHs will only take place if a PAH-DNA adduct is formed (Höner 2001; Huang and Penning 2014). Developmental toxicity of PAHs occurs because PAHs have lipophilic characteristics enabling them to reach the embryo and fetus by passing through the placental barrier of a pregnant woman (Herbstman et al. 2012). In addition, immunotoxic effects from the PAHs are highly associated with immunosuppression where the exposed individual will have a higher susceptibility to infectious diseases and the formation of cancers (Huang and Penning 2014).

The International Agency of Research on Cancer (IARC) has categorized PAHs into five groups: Group 1 (carcinogenic to humans), Group 2A (probably carcinogenic to humans), Group 2B (possibly carcinogenic to humans), Group 3 (not classifiable regarding its carcinogenicity towards humans), and Group 4 (probably not carcinogenic to humans). BaP is one of the most carcinogenic compounds and has been regrouped from Group 2A to Group 1 and CHR has been reclassified from Group 3 to Group 2B, whereas BaA was reassigned from 2A to 2B (Ishizaki et al. 2010).

Sources and Occurrence of PAHs

PAHs are ubiquitous and diffusion into the environment can occur easily through both natural events and man-made processes. The World Health Organization (WHO 1998) stated that the primary anthropogenic products involved are from

petroleum refineries, industrial machinery manufacturing, motor vehicle exhausts, coke production, barbeque smoke, tobacco smoke, and etc. Considerable amounts of PAHs are also found to be generated from natural sources, for instance, volcanic activity and forest fires. Furthermore, PAHs can be found in water (Rey-Salgueiro et al. 2009; Zhang et al. 2012), soil (Shang et al. 2014; Yu et al. 2019), air (Lammel et al. 2010), foods (García-Falcón et al. 2005a; Yebra-Pimentel et al. 2012), and street dust (Lorenzi et al. 2011; Gope et al. 2018). Consequently, the careful monitoring of PAHs is vital as certain PAHs have been found to be carcinogens in experimental animals, indicating the possible carcinogenic effects on humans (ATSDR 2009).

Occurrence of PAHs in Food

Natural sources and environmental pollution such as the deposition of airborne particulates to the soil and surface of plants can lead to the contamination of food with PAHs (Killian et al. 2000; Smith et al. 2001). Research has discovered that around 79–99% of mankind's susceptibility to PAHs were contributed by food consumption and it has been estimated that the possible total intake of PAHs through food is 2.5 µg/day (Menzie et al. 1992). Roasting, toasting, baking, smoking, frying, and baking are some of the food preparation techniques that can cause the formation of PAHs (Codex Alimentarius Commission 2005; European Commission 2002; Perelló et al. 2009). Moreover, PAHs have been found in food following preservation processes such as traditional drying and curing of food (de Vos et al. 1990). Various food samples, including roasted coffee (Houessou et al. 2006; Guatemala-Morales et al. 2016), tea (Lin et al. 2005; Wu et al. 2020), milk (Aguinaga et al. 2008; Sun et al. 2020), alcoholic beverages (Menezes et al. 2015; Singh et al. 2016), oils (Rodríguez-Acuña et al. 2008; Yousefi et al. 2018), smoked meat (Jira et al. 2008; Zachara et al. 2017), smoked cheese (Guillén and Sopelana 2005; Gul et al. 2015), smoked fish (Lund et al. 2009; Mahugija and Njale 2018), fruits (Jánská et al. 2006; Paris et al. 2018), and vegetables (Jánská et al. 2006; Jia et al. 2018) were found to contain PAHs at µg/kg concentrations.

The Scientific Opinion of the Panel on Contaminants in the Food Chain indicated that BaP alone was insufficient to gauge the amount of PAHs present in food (Alexander et al. 2008). Therefore, they suggested using PAH2, PAH4, and PAH8 to estimate the margins of exposure (MOE). PAH2 indicates BaP and CHR, whereas, PAH4 means BaP, BaA, Chry, and BbFA and PAH8 includes BaP, BaA, BkFA, CHR, BbFA, DBahA, BghiP, and IP (Alexander et al. 2008).

PAHs Found in Beverages

In the past 16 years, extensive research studies have been carried out by scientists to analyze the amount of PAHs in various beverages such as tea, coffee, milk, and alcoholic beverages as PAHs are formed during their production processes. The following sections provide a summary of the sources of PAHs in these beverages.

Tea

Tea is the second most consumed non-alcoholic beverage, the first being water (Benson et al. 2018). The Food and Agriculture Organization (FAO) of the United Nations (FAO 2012) stated that around 4 million tonnes of tea were consumed in 2010 and in the last 10 years; the quantity of tea produced has increased to an approximate of 5.35 million tonnes in 2013 (FAO 2015). Tea is known to be refreshing, medicinally beneficial, and an antioxidant, as well as having mild stimulant properties and pleasant aromas (Kuroda and Hara 1999; Pittler 2005). Agricultural products including raw ingredients of tea have a likelihood of being contaminated with contaminants such as PAHs. An example would be PAHs that are in the gaseous state or PAHs that are bounded to particulates in the air, which could be easily deposited on the leaves of tea and hence causing accumulation to occur (Jánská et al. 2006). Moreover, the accumulation of hydrophobic PAHs in fruit and herbal teas is possible due to the presence of lipophilic compounds, such as essential oils (Schlemitz and Pfannhauser 1997). PAHs are produced during the fresh leaf treatment process, such as the burning of oil, coal, and wood for drying or roasting of tea leaves (Lin et al. 2005). The analysis of PAHs in tea can be quite challenging as tea contains various co-extractives, for example, high levels of chlorophyll, polyphenols, organic acids, caffeine, sugars, and pigments. Nevertheless, the concentrations of co-extractives are highly dependable on the types of tea. Black teas are considered to have a complicated matrix as there is a high amount of interfering substances. The EU Commission (2011) has established regulated maximum levels of allowable PAHs on various food products, but none have been accepted and acknowledged for teas yet. Nevertheless, the maximum contaminant level (MCL) of PAHs in drinking water was set to be as follows: (1) 0.1 ppb for BaA, (2) 0.2 ppb for BaP, BbFA, BkFA, CHR, (3) 0.3 ppb for DBahA, and (4) 0.4 ppb for IP (ATSDR 2009b).

Coffee

Coffee is widely consumed around the world and chemical substances such as proteins, carbohydrates, fats, vitamins, water, minerals, flavoring substances, organic acids, and

caffeine are present in coffee beans (Lee and Shin 2010). Roasting is an important step in order to bring out the flavor, aroma, and color of coffee and is normally carried out with temperatures of 120–230 °C (Tfouni et al. 2012). On the other hand, roasting can also prompt the production of unwanted and hazardous compounds such as PAHs, acrylamide, and furan (Tfouni et al. 2013). Additionally, coffee drinks that were brewed with PAH-contaminated green coffee beans might contain PAHs as well (Houessou et al. 2008). Nevertheless, for coffee and coffee substitutes, no maximum levels of PAHs have been established by the European Union (EU) Commission (Sadowska-Rociek et al. 2015).

Milk

Milk is an important essential dairy product required by humans and it contributes to the dietary intake of zinc, vitamin B12, magnesium, calcium, pantothenic acid, and selenium (Raza and Kim 2018). In dairy farming process, animal feed are at risk of contamination from water, soil, and air; organic contaminants such as PAHs can be transferred from the animals that have fed on contaminated feeds through different routes such as milk, urine, and feces (Grova et al. 2002). PAHs that are ubiquitous in the environment and are lipophilic have a huge tendency to be distributed in milk (Zhao et al. 2017, 2018). Hence, human diets will be contaminated with PAHs upon the ingestion of milk containing PAHs.

Various milk samples undergo processes such as pasteurization and ultra-high-temperature processing (UHT). Results have shown that all the raw milk samples analyzed contained PAHs, but higher concentrations were observed in the UHT and pasteurized milk. This indicates that these heat treatment procedures have an effect on raising the levels of PAHs in milk (Naccari et al. 2011).

Alcoholic Beverages

Spirits are high-degree alcoholic beverages with an average alcohol content of 40% v/v and they are obtained from the distillation of low-degree alcoholic beverages (alcohol which are produced from the fermentation of sugar) (Cacho et al. 2016). Some examples of spirits are cachaça, gin, vodka, whisky, brandy, and rum. The WHO (2014) reported that around 9.2 L of pure alcohol is consumed per capita per year in the USA. However, researchers have discovered that PAHs can contaminate some of the spirits through various manufacturing processes. Cachaça is a Brazilian alcoholic distillate manufactured from fermented sugar cane juice and is ranked as the third largest distilled spirit consumed worldwide (Cardoso et al. 2004). Before the harvesting of the sugar canes take place, the sugar canes are being burnt to

evaporate off the water in the stalk and to increase the sugar weight. This can contribute to the production of PAHs as the incomplete combustion of organic matters occur during this process and may be attached to the stalks of the sugar canes, hence contaminating the sugar cane juice during processing (Galinaro et al. 2007).

Rum is commonly produced from the distillation of fermented sugar cane molasses (by product of refining sugar cane) or sugar cane juice. Similar to the cachaça production process, the sugar cane is first burnt which creates the main source of contamination for PAHs (Riachi et al. 2014). In contrast, the flavor of whisky is developed during the aging process which involves smoking or charring oak wooden casks. This can lead to the migration of PAHs present in the woods to the whisky (Chinnici et al. 2007; Da Porto et al. 2006).

The production of cereal-based beverages such as beer, which is the most commonly consumed alcoholic beverage in the world, involves the roasting and toasting of barley, which are the main source of PAHs (Anderson et al. 2019; Rascón et al. 2019). Wine is commonly aged by being stored inside wood barrels or in steel tanks containing wood chips (Rascón et al. 2019). Chinnici et al. (2007) reported that the wood of the barrels and chips is naturally aged by the weather for 1–3 years, but are also toasted with open oak fires and other methods of toasting (electrical and convective heating), which will generate a significant amount of PAHs.

Analytical Methods for PAH Determination

Different methods have been validated for the extraction of PAHs in accordance with the matrixes of each food item since there is no existence of standardized methods for a particular matrix. Multiple extractions steps together with preconcentration procedures need to be carried out for PAHs that are in the ppb levels. In addition, there are challenges in obtaining repeatability for PAHs with two or three aromatic rings. Therefore, rapid, simple, and dependable analytical methods need to be developed so that precise deduction of contaminants in foods can be achieved. From this, the actual exposure of humans to PAHs in food can be evaluated.

Sample Preparation

Prior to the PAH analysis of samples, PAH samples must be protected against oxidation and photoirradiation processes as PAHs are sensitive towards light (Skláršová et al. 2012). Hence, minimum exposure of light towards the samples during matrix pretreatment is highly encouraged. Tamakawa et al. (1992) has found that during the sample preparation to extract PAHs, three or fewer aromatic rings of PAHs (ANT, FA and PY) were found to be sublimated easily during the

concentration processes. Therefore, concentration to dryness should be monitored carefully to minimize any evaporation of PAHs and minimize losses of lower molecular weight PAHs. Internal standards or surrogates are being advised to be added to the samples before extraction to ensure precise and accurate quantification by analytical instruments (Guillen et al. 2000a, 2000b, 2000c).

Extraction

Various extraction methods have been proposed and validated by researchers globally; however, only successful and effective extraction procedures are integrated into the experiments to ensure that samples have minimum analytical interferences. Alkaline saponification/solvent extraction using ethanolic KOH, NaOH, and methanolic solution is the most common method used for the removal of fats, pigments, and other organic contaminants to avoid any interference during the analysis (Tatatsuki et al. 1985; Girelli et al. 2017). Certain PAHs can easily undergo chemical, physical, and biological change or breakdown when harsh saponification conditions are applied; therefore, for food samples that have low content of fat, liquid–liquid extraction (LLE) can be applied (Tatatsuki et al. 1985). Newer methods such as the QuEChERS method (abbreviated from quick, easy, cheap, effective, rugged, and safe) (Pincemaille et al. 2014) and dispersive liquid–liquid microextraction (dLLME) (Rivera-Vera et al. 2019) have been developed and diligently used by scientists for the determination of PAHs. In addition, the two newer methods mentioned above are more cost efficiency and safer as less solvent will be consumed during the process of extraction (Zelinkova and Wenzl 2015; Purcaro et al. 2013).

Other methods for PAH extraction in beverages are pressurized liquid extraction (PLE) (Ziegenhals et al. 2008), ultrasound-assisted extraction (UAE) (Guatemala-Morales et al. 2016), solid-phase extraction (SPE) (Caruso and Alaburda 2009), Soxhlet extraction (Grover et al. 2013), solid-phase microextraction (SPME) (Viñas et al. 2007), stir bar sorptive extraction (SBSE) (Zuin et al. 2005), membrane-assisted solvent extraction (MASE) (Mañana-López, et al. 2021), microwave-assisted extraction (MAE) (Kamallabadi et al. 2018), and magnetic solid-phase extraction (MSPE) (Shariatifar et al. 2020). However, due to the challenging matrices present, inconsistent recoveries are often obtained and therefore causing an interference to the peaks in the chromatograms. The advantages and disadvantages of each method mentioned above are summarized in Table 1.

For tea samples, the commonly used method for extracting PAHs from the matrix is QuEChERS (Sadowska-Rociek et al. 2014; Tfouni et al. 2018). Recently, modern analytical

chemistry strives to develop and integrate green chemistry into their research in which there is reduced usage of samples and solvents, which also employ easy procedures, simple analytical equipment, and reagents that are not harmful to individual health and the environment (Sadowska-Rociek et al. 2014). One of them is QuEChERS which was initially developed for the analysis of pesticide residues in food samples of plant origin (Anastassiades et al. 2003). On the other hand, coffee samples were conventionally extracted by saponification (Lee and Shin 2010). This is because coffee contains around 15% lipids which can cause interference during the analysis of PAHs (Houessou et al. 2006); hence, saponification using ethanolic KOH, NaOH, and methanolic solution is an effective method used for lipid removal. Alkaline saponification followed by LLE is also commonly done to remove fatty acids present in the matrix (Lee and Shin 2010). Chung et al. (2010) and Naccari et al. (2011) have applied saponification to extract PAHs from milk samples. This is due to the fact that PAHs are lipophilic and have a huge tendency to be distributed in milk (Zhao et al. 2017, 2018). According to literature, various methods ranging from LLE, SPE, SPME, QuEChERS, UAE, and DLLME were used to extract PAHs from alcoholic beverages (Will et al. 2018). The LLE and SPE were the preferred methods many years ago, but in recent years, SPME, DLLME, and QuEChERS methods were used as analytical extraction methods for alcoholic beverages (Galinaro et al. 2007; Da Silva et al. 2019).

Cleanup

The cleanup of extracts is carried out to separate target PAHs from other compounds and to remove any interferences that can make the determination of PAHs challenging. Various cleanup methods such as column chromatography, SPE, gel permeation chromatography (GPC), and thin-layer chromatography (TLC) have been studied extensively (Tamakawa 2008). Normally, large volumes of hazardous solvents need to be used to purify the PAHs because they have lipophilic properties and their extraction is correlated with lipid constituents of food (Purcaro et al. 2013).

On the other hand, commercial SPE cartridges have substituted conventional chromatographic methods and are widely used as the cleanup step for the purification of PAHs in water samples (Kouzayha et al. 2011), food (Bishnoi et al. 2005), and airborne particles (Tala and Chantara 2019), due to their multiple advantages such as higher recoveries obtained, less time consumption, and reduced usage of hazardous solvents (Sibiya et al. 2012). Additionally, SPE is excellent not only for sample cleanup, but also for sample extraction and concentration (Sibiya et al. 2012).

Table 1 Advantages and disadvantages of different extraction methods

Extraction methods	Principles	Advantages	Disadvantages	References
Supercritical fluid extraction (SFE)	This method utilizes supercritical fluid such as carbon dioxide which is above the critical temperature and pressure as an extraction medium	<ul style="list-style-type: none"> ❖ No usage of organic solvents and its environmentally safe and inexpensive ❖ Faster extractions ❖ Automations are possible in extractions 	<ul style="list-style-type: none"> ❖ Limitation for polar products ❖ Needs complicated apparatus ❖ Uses elevated pressure 	Knez et al. 2010
Microwave-assisted extraction (MAE)	Microwave energy use to heat the extracting solvent that is in contact with the sample and extract the analytes of interest from the sample into the solvent	<ul style="list-style-type: none"> ❖ Reduced extraction time (< 30 min) ❖ Less solvent consumption (10–40 mL) ❖ Improved extraction yield and product quality 	<ul style="list-style-type: none"> ❖ Microwaves will under extract with the presence of non-polar or volatile-targeted compounds ❖ Usage of elevated temperatures may lead to heat-sensitive bioactive compounds being degraded 	De la Guardia and Armenta 2011 Veggi et al. 2013
Pressurized liquid extraction (PLE)	This method operates by the extraction with organic solvents at high temperatures and pressure so that the extractant will remain in the condensed state	<ul style="list-style-type: none"> ❖ Automated system ❖ Solvent consumption are less (10–100 mL) ❖ Shorten extraction times (< 30 min) 	<ul style="list-style-type: none"> ❖ Expensive maintenance ❖ Cleanup is needed 	Ridgway et al. 2012
Ultrasound-assisted extraction (UAE)	This method uses ultrasonic waves for extraction. UAE can be used in the pretreatment step to improve the efficiency of extraction processes	<ul style="list-style-type: none"> ❖ Less energy and solvent consumption ❖ Extraction time are reduced ❖ Lower costs are needed ❖ Environmentally friendly 	<ul style="list-style-type: none"> ❖ Limitation of large-scale application because of more expensive costs 	Sasidharan et al. 2018 Rani et al. 2020
Dispersive liquid–liquid microextraction (DLLME)	Injection of fine droplets of disperser solvent into an aqueous sample solution to form a cloudy solution, then the mixture is centrifuged which forms aqueous phase (upper) and sedimented phase (lower). The supernatant is disposed and the sedimented phase is taken up by syringe and analyzed	<ul style="list-style-type: none"> ❖ Easy to use ❖ Inexpensive, fast, and simple ❖ High recovery and high enrichment factor obtained ❖ Automation is possible 	<ul style="list-style-type: none"> ❖ Limitations on the selection of extraction solvent ❖ Three solvents are needed 	Quigley et al. 2016 Rodrigues et al. 2019
Liquid–liquid extraction (LLE)	This method also known as partitioning or solvent extraction is carried out by the selectively extraction of a certain analyte based on their partitions between two non-miscible liquid phases (organic and aqueous)	<ul style="list-style-type: none"> ❖ Simple operation ❖ Simple apparatus 	<ul style="list-style-type: none"> ❖ Costly, high purity, and potential hazardous organic solvents are used ❖ Decrease in sensitivity for volatile analytes ❖ Emulsion formation 	Tamakawa 2004 Kim et al. 2012

Table 1 (continued)

Extraction methods	Principles	Advantages	Disadvantages	References
S Soxhlet extraction	This technique is usually applied to analytes that are adequately thermally stable. Boiling and condensation occur continuously when the solvent (extractant) repeatedly cycled through the sample and the sample extracted is being collected in the hot solvent in round bottom flasks	<ul style="list-style-type: none"> ❖ A simple method ❖ Ability to extract more sample mass than MAE and SPE 	<ul style="list-style-type: none"> ❖ Long extraction time is needed ❖ Usage of heat may not be that suitable for thermolabile compounds ❖ The formation of artifacts due to long period of time exposure to organic chemicals 	Tamakawa 2004 Ridgway et al. 2012
Solid-phase extraction (SPE)	This method use cartridges containing solid adsorbent such as silica, C18, etc.to absorb the desired analyte from the matrix	<ul style="list-style-type: none"> ❖ Minimize solvents usage ❖ Selectivity derived from sorbent choice and the elution procedure followed 	<ul style="list-style-type: none"> ❖ SPE cartridges and disks susceptible to plugging ❖ Only dissolved PAHs extracted 	Berrueta et al. 1995 Tamakawa 2004
Solid-phase microextraction (SPME)	A microsampling method that is fast, solvent-free, and allows sampling, isolation, and enrichment in a single step	<ul style="list-style-type: none"> ❖ Solvent-free technique ❖ Can be automated ❖ Simple and fast 	<ul style="list-style-type: none"> ❖ Lack of quantitative results when recoveries were tested ❖ Short lifetime of the SPME fibers 	Padilla-Sánchez et al. 2013
Dispersive solid-phase extraction (d-SPE)	A method where solid sorbents are being dispersed in liquid samples (matrix). The close proximity of the sorbent and analyte of interest will enhance the sorption process, hence, improving the efficiency of the extraction	<ul style="list-style-type: none"> ❖ More contact of sorbents with analyte which enhances the retention of analyte to the sorbents ❖ Less usage of solvent and sorbents ❖ More economical 	N/A	Islas et al. 2017 Chisvert et al. 2019
Magnetic solid-phase extraction (MSPE)	A form of d-SPE where magnetic nanoparticles (MNPs) are used as adsorbents to adsorb the target analytes from complex matrices	<ul style="list-style-type: none"> ❖ Shortens duration time of analysis by reducing the amount of steps in the extraction process ❖ Less usage of organic solvents and more environmentally friendly 	<ul style="list-style-type: none"> ❖ In biological samples, it can be quite difficult to isolate small amount of target analytes from complex matrices ❖ Non-specific binding can caused an increase in background signals 	Wierucka and Biziuk 2014 Manousi et al. 2020
QuEChERS	The main principle of QuEChERS involves liquid–liquid partitioning with solvent (usually acetonitrile) and purification of the extract will be done using d-SPE	<ul style="list-style-type: none"> ❖ Simple and fast ❖ Less solvent usage ❖ Cheaper than conventional SPE 	<ul style="list-style-type: none"> ❖ For samples that have high fat content, the extraction efficiency is low and the purification process will cause a huge loss 	Cunha et al. 2010 Hawach scientific 2010 Rejczak and Tuzimski 2015

Table 1 (continued)

Extraction methods	Principles	Advantages	Disadvantages	References
Stir bar sorptive extraction (SBSE)	SBSE is a method based in sorptive extraction in which the solutes are extracted into a magnetic stirring rod coated with polydimethylsiloxane phase (polymer). This method also utilizes sample preparation steps without any usage of solvent for the extraction and enrichment of organic compounds from aqueous matrices	<ul style="list-style-type: none"> ❖ Huge amount of polydimethylsiloxane on the surface of stir bar will enhance the recovery of the targeted analytes as well as its sensitivity ❖ SBSE can be applied in variety of analytical fields such as food, environmental and clinical analysis 	<ul style="list-style-type: none"> ❖ Limited spectrum of analyte polarities for the available stationary phases ❖ Strong matrix effects 	David and Sandra 2007 Camino-Sánchez et al. 2014 Telgheder et al. 2018
Membrane assisted solvent extraction (MASE)	This method utilizes hydrophobic and size exclusive polymeric microporous membranes that allow the sample to be separated from the solvent (acceptor phase)	<ul style="list-style-type: none"> ❖ Less solvents used compared to LLE ❖ High enrichment factors can be obtained ❖ MASE setup can be integrated with ease into a flow system and automated for on-line coupling to GC, HPLC, and capillary electrophoresis 	<ul style="list-style-type: none"> ❖ Organic solvents used are quite toxic (even though only small volumes) ❖ This method can only analyze liquid or gaseous samples 	Mañana-López et al. 2021

N/A not available

Table 2 Preparation procedures and GC conditions used for the analysis of PAHs in beverages

Sample	PAH	Sample treatment and prepreparation	Analytical instrument; column (column dimension)	Oven temperature program	Injection mode/temperature	LOD (LOQ)	Percentage recovery (%)	Article
Tea	15 + 1 EU PAHs ^a	PLE (acetonitrile and hexane) followed by GPC and SPE	Fast-GC/HRMS; TR-50MS (10 m × 0.1 mm, 0.1 µm)	140 °C (1 min) to 240 °C (10 °C/min) to 270 °C (5 °C/min) to 280 °C (30 °C/min) to 290 °C (4 °C/min) to 315 °C (30 °C/min) to 330 °C (3 °C/min)	Splitless (320 °C)	0.01–0.02 µg/kg	75–117	Ziegenhals et al. (2008)
Tea	BaP, BaFA, ANT	MAE with DMSO (dimethyl sulfoxide) then extracted with n-hexane	GC-MS; DB-5MS (30 m × 0.25 mm, 0.25 µm)	100 °C (2 min) to 180 °C (10 °C/min, 5 min) to 280 °C (15 °C/min, 10 min)	Splitless (250 °C)	0.18–3.58 µg/kg	77.1–116	Li et al. (2011)
Tea	15 + 1 EU PAHs ^a	Modified QuEChERS method for extraction Cleanup with SPE on molecularly imprinted polymers (MIPs)	GC × GC-TOFMS First column: BPX-50 (30 m × 0.25 mm, 0.25 µm) Second column: BPX-5 (1 m × 0.1 mm, 0.1 µm)	First dimension: 80 °C (4.3 min) to 220 °C (30 °C/min) to 240 °C (2 °C/min, 0 min), to 360 °C (10 °C/min 15 min) second dimension: temperature offset 10 °C; carrier gas helium with a ramped flow 1.3 mL/min (19 min) at 50 mL/min to 2 mL/min (16 min)	Split/splitless	LOQ: 0.05–0.20 µg/kg	73–103	Drabova et al. (2012)
Dried tea	US EPA 16 PAHs	Subtle extraction and two purification steps by passing through a column packed with anhydrous Na_2SO_4 and a silica gel column	GC-FID; ZB-5MS (30 m × 0.25 mm, 0.25 µm)	60 °C (4 min) to 320 °C (6 °C/min, 10 min) to 321 °C (1 °C/min, 1 min)	Split ratio of 1:3 (275 °C)	N/A	N/A	Grover et al. (2013)
Smoked, non-smoked teas, and tea infusions	PAH4	QuEChERS method: Two SPE purification steps: (1) first step on a C ₁₈ support (trap the non-polar compounds) and second step on a polar support (Florisil) to trap polar impurities	GC-MS/MS; HP-5MS (30 m × 0.25 mm, 0.25 µm)	70 °C (5 min) to 200 °C (10 °C/min, 0.5 min) to 244 °C (5 °C/min, 0.5 min) to 266 °C (5 °C/min, 0.5 min) to 300 °C (5 °C/min, 3.5 min. Total run time was 48 min)	N/A	Tea leaves: 0.2–0.3 µg/kg (0.5–0.6 µg/kg) Tea infusions: 100 ng/L (200–400 ng/L)	Tea leaves: 53–82 Tea infusions: 67–88	Pinecaille et al. (2014)
Dried tea and tea infusions	15 + 1 EU PAHs ^a	For dried teas: Supercritical then cleanup with automated SPE (polystyrene-divinylbenzene phase) For infusions: LLE (cyclohexane) and cleanup with SPE (polystyrene-divinylbenzene phase)	GC-MS; Select PAH (15 m × 0.15 mm, 0.10 µm)	70 °C (0.4 min) to 180 °C (70 °C/min, 1 min) to 280 °C (7 °C/min, 7 min) to 300 °C (50 °C/min, 7 min) to 350 °C (30 °C/min, 3.6 min)	Splitless	LOQ: (0.5–1.0 µg/kg)	71–128	Schulz et al. (2014)
Tea	US EPA 16 PAHs excluding NPH, ACP, FA, BghiP	Modification of QuEChERS method: Acetonitrile used as the extraction solvent + SAX + PSA as sorbents and LLE	GC-MS; ZB MR 1 (30 m × 0.25 mm, 0.25 µm)	50 °C (1.0 min) to 320 °C (15 °C/min, 6.0 min)	Splitless (270 °C)	0.2–0.4 µg/kg (0.6–1.2 µg/kg)	50–120	Sadowska-Bociek et al. (2014)
Tea infusions	US EPA 16 PAHs	Extracted using UAE and cleanup using SPE (2 g of silica gel)	GC-FID; HP-5 (30 m × 0.25 mm, 0.25 µm)	60 °C (1 min) to 300 °C (20 °C/min, 10 min)	Splitless (250 °C)	0.03–0.24 µg/kg (0.1–0.8 µg/kg)	64.1–99.4	Iwegbue et al. (2016)
Fresh green tea and tea infusions	US EPA 16 PAHs excluding NPH	Green tea leaves: Extracted with modified QuEChERS method with acetonitrile + MgSO ₄ + NaCl + PSA + Florisil + C ₁₈ Tea infusions: extracted using LLE (n-hexane)	GC-MS/MS; VF-5 MS (30 m × 0.25 mm, 0.25 µm)	50 °C (5 min) to 200 °C (15 °C/min, 2.0 min) to 280 °C (5 °C/min, 12 min)	Splitless (250 °C)	Tea leaves: 1–4 µg/kg (3.2–13.3 µg/kg) Tea infusions: 23.1–97.1 ng/L (74.5–323.3 ng/L)	72.0–111.5	Gao et al. (2017)
Dry tea	US EPA 16 + Carbazole (CBZ)	Extracted using LLE (n-hexane) and purification done by column chromatography on silica gel	GC-FID; HP-5 (30 m × 0.32 mm, 0.25 µm)	50 °C (0.4 min) to 195 °C (20 °C/min, 3.0 min) to 250 °C (8 °C/min, 5.0 min) to 290 °C (5 °C/min, 1.0 min)	N/A	0.3 µg/kg (0.9 µg/kg)	90.24–108.92	Benson et al. (2018)
Tea infusions	US EPA 16 PAHs + α ^b	Tea samples: Extracted using acetone:hexane (1:1 V/V) then GPC Tea infusions: Acidified with formic acid, LLE (n-hexane) and clean-up using silica gel column	GC-MS/MS; Rtx-17 MS fused-silica (20 m × 0.18 mm, 0.18 µm)	30 °C (2 min) to 180 °C (15 °C/min) to 190 °C (2 °C/min) to 290 °C (2.5 °C/min to 320 °C (5 °C/min, 15 min)	Split mode	0.01–0.09 µg/kg (0.02–0.32 µg/kg)	70–120	Rosko et al. (2018)
Tea infusions	US EPA 16 PAHs + 1-MN (1-Methylnaphthalene), 2-MN (2-Methylnaphthalene)	QuEChERS extraction: MgSO ₄ + NaCl + PSA + C ₁₈	GC-MS/MS; DB-5MS (30 m × 0.25 mm, 0.25 µm)	70 °C (1 min) to 270 °C (10 °C/min) to 280 °C (2 °C/min, 3 min) to 310 °C (2 °C/min, 1 min)	N/A	10–200 ng/L (30–600 ng/L)	71–110	Tinn-Lam et al. (2018)
Tea infusions	US EPA 16 PAHs	MSPE were carried out with Fe ₃ O ₄ @SiO ₂ -C ₆₀ as the composite	GC-MS; HP-5MS (30 m × 0.25 mm, 0.25 µm)	80 °C (1 min) to 230 °C (25 °C/min, 6 min) to 300 °C (10 °C/min, 6 min)	Splitless (280 °C)	0.8–14.3 ng/L (2.6–47.6 ng/L)	92.4–106.7	Zhou et al. (2018)

Table 2 (continued)

Sample	PAH	Sample treatment and prepreparation	Analytical instrument; column (column dimension)	Oven temperature program	Injection mode/temperature	LOD (LOQ)	Percentage recovery (%)	Article
Dry tea and infusions	US EPA 16 PAHs	Dried teas: Ultrasonication (hexane) and cleanup with SPE (1 g of Florisil)	GC-MS; ZB-5MS (30 m×0.25 µm, 250 µm)	80 °C (0.5 min) to 230 °C (10 °C/min, 5 min), 5 min to 300 °C (5 °C/min, 15 min)	Splitless (250 °C)	Dried teas: 0.09–0.32 µg/kg (0.28–0.97 µg/kg) Tea infusions: 0.1–0.28 ng/L (0.29–0.86 ng/L)	Dried teas: Low spike (10 µg/kg): 37.10–88.24 High Spike (50 µg/kg): 37.0–96.10 Tea infusions: Low spike (500 ng/L): 35.53–78.73 High spike (2000 ng/L): 38.33–104.7	Cierniak et al. (2019)
		Tea infusions: LLE (cyclohexane) and cleanup done on SPE column filled with 1 g of Florisil						
Dry tea	PAH8	UAE (n-hexane) and purified using column chromatography on silica gel	GC-FID; HP-5 (30 m×0.32 mm, 0.25 µm)	50 °C (0.4 min) to 195 °C (20 °C/min, 3.0 min) to 250 °C (8 °C/min, 5.0 min) to 290 °C (5 °C/min, 1.0 min)	N/A	N/A	PHE: 90.24 DBahA: 108.92	Fred-Ahmadu and Benson (2017)
Tea infusions	US EPA 16 PAHs	MSPE was carried out with magnetic-MWCNT (multi-walled carbon nanotubes) composite	GC-MS; DB-5MS (30 m×0.25 mm, 0.25 µm)	70 °C (1 min), to 300 °C (10 °C/min, 7 min)	Splitless (290 °C)	0.040–0.084 µg/kg (0.120–0.252 µg/kg)	91.1–102.3	Roudsari et al. (2020)
Dry tea and tea infusions	US EPA 16 priority PAHs excluding NPH	Dried teas: Extracted with DCM and there are two cleanup steps: 1.) GPC (50 cm length×2 cm ID) 2.) column chromatography on silica gel (5 g) Tea infusions: LLE (DCM) and purification using column chromatography on silica gel (5 g)	GCMS-QP2010; BPX5 (60 m×0.25 mm, 0.25 µm)	60 °C 2 min) to 210 °C (30 °C/min) to 310 °C (5 °C/min, 15 min)	N/A	For PAH4 <0.3 µg/kg (<0.9 µg/kg)	53–132	Phan Thi et al. (2020)
Dry tea leaves and tea infusions	15 + 1 EU PAHs ^a	Dry tea: UAE (n-hexane/dichloromethane), alkaline saponification then cleanup using SPE (polystyrene-divinylbenzene) Tea infusions: UAE (n-hexane) and concentrated to dryness using rotary evaporator	GC-Qq-MS DB-EUPAH column (20 m×0.18 mm, 0.14 µm)	80 °C (2 min) to 250 °C (10 °C/min, 2 min) to 315 °C (8 °C/min, 5 min) to 320 °C (20 °C/min, 8 min)	Splitless (250 °C)	0.3 µg/kg (1.0 µg/kg)	92.4–101.4	Wu et al. (2020)
Coffee brew	US EPA 16 PAHs exclude NPH + etc ^a	Saponification with potassium hydroxide-methanol solution and LLE (hexane)	GC-MS; Equity 5 fused silica (30 m×0.5 µm, 0.25 µm)	40 °C (2 min) to 100 °C (40 °C/min) to 200 °C (10 °C/min) to 325 °C (30 °C/min, 8 min)	Splitless (280 °C)	0.67–18 ng/L	> 85	Onecchio et al. (2009)
Coffee	PAH4	PLE and cleanup up using GPC and SPE (silica)	GC-MS; Select PAH (15 m×0.15 mm, 0.1 µm)	70 °C (1 min) to 180 °C (60 °C/min) to 220 °C (4 °C/min, 10 min) to 280 °C (28 °C/min, 10 min) to 340 °C (14 °C/min, 5 min)	N/A	0.1–0.3 µg/kg	94–106	Diedahl-Olesen et al. (2015)
Ground coffee	PAH8 + FA, PY	PLE then LLE and cleanup up with silica columns	GC-MS; DB-5MS (30 m×0.25 mm, 0.25 m)	70 °C (1 min) to 160 °C (40 °C/min) to 300 °C (6 °C/min)	Splitless (260 °C)	0.03–0.18 µg/kg	87–111	Pissinatti et al. (2015)
Ground, instant and cereal coffee	PAH4	1). QuEChERS where d-SPE with primary secondary amine (PSA), strong anion exchange (SAX) sorbents and NH_2 2). LLE UAE (n-hexane and methylene chloride) then alkaline saponification and cleanup with SPE (1 g of packed silica)	GC-MS; DB-5MS (30 m×0.25 mm, 0.25 µm)	50 °C (1.0 min) to 320 °C (15 °C/min, 6.0 min)	Split/splitless (270 °C)	0.18–0.33 µg/kg (0.6–1.1 µg/kg)	70–104	Sadowska-Rociek et al. (2015)
Ground coffee	16 US EPA PAHs	Extracted with MAE (KOH) and DLLME	GC-MS; HP-5MS (30 m×0.25 mm, 0.25 µm)	40 °C (1 min) to 110 °C (30 °C/min) to 300 °C (5 °C/min) to 310 °C (20 °C/min, 3.2 min)	Splitless	0.38–2.6 µg/kg (1.27–7.20 µg/kg)	45–144	Guatemala-Morales et al. (2016)
Coffee	PAH2 + BaA, BbFA, IP, B[a]FL (benzo[e]fluorene) and 5-MCH (5-methylchrysene)		GC-MS; HP-5MS (30 m×0.25 mm, 0.25 µm)	100 °C (1 min) to 180 °C (10 °C/min, 5 min) to 250 °C (5 °C/min, 5 min) to 300 °C (4 °C/min, 20 min)	Splitless (290 °C)	0.1–0.3 µg/kg (0.3–0.9 µg/kg)	88.1–101.3%	Kamalabadi et al. (2018)
Milk and milk powders	16 US EPA PAHs	Direct immersion-solid phase microextraction (DI-SPME)	GC-MS; HP-5MS (30 m×0.25 mm, 0.25 µm)	40 °C to 120 °C (10 °C/min) to 300 °C (3 °C/min, 2 min)	Splitless (270 °C)	3–1500 ng/L	87.6–112%	Agutunga et al. (2007)
Milk	16 US EPA PAHs	Saponification carried out in LLE, then mix dichloromethane and n-hexane (4:1, v/v) and purification of the organic layers	GC-MS; HP-5MS (30 m×0.25 mm, 0.25 µm)	65 °C (1 min) to 150 °C (20 °C/min) to 317 °C (6 °C/min, 8 min)	N/A	N/A	80–120	Chung et al. (2010)
Milk	16 US EPA PAHs	Fat was extracted using detergent solution in warm water bath Purified by column chromatography on silica gel	GC-MS; HP-5 (30 m×0.35 mm, 0.25 µm)	90 °C to 180 °C (8 °C/min, 1 min) to 245 °C (5 °C/min) to 300 °C (2 °C/min)	Splitless (320 °C)	10–100 µg/kg	50–95	Guárcel et al. (2015)

Table 2 (continued)

Sample	PAH	Sample treatment and preservation	Analytical instrument; column (column dimension)	Oven temperature program	Injection mode/temperature	LOD (LOQ)	Percentage recovery (%)	Article
Milk and milk powders		QuEChERS extraction and cleanup using normal phase mode of SPE	GC-MS/MS; HP-5MS (30 m × 0.25 mm, 0.25 µm)	70 °C to 250 °C (15 °C/min) to 315 °C (5 °C/min, 5 min)	N/A	20–42 µg/kg	N/A	Rawash et al. (2018)
Milk	16 US EPA PAHs excluding NPH, ACP, FA	MSPE was carried out with multi-walled carbon nanotubes-Magnetic nanoparticles (MWCNT-MNP) composite	GC-MS; DB-5MS (30 × 0.25 mm × 0.25 mm)	70 °C (1 min) to 300 °C (10 °C/min, 7 min)	Splitless (290 °C)	0.040–0.075 µg/kg (0.121–0.227 µg/kg)	86.1–100.3	Shariatifar et al. (2020)
Milk	16 US EPA PAHs	QuEChERS extraction and EMR-lipid used for pretreatment step	GC-QQ-MS; DB-5MS (30 m × 0.25 mm, 0.25 µm)	80 °C (2 min) to 140 °C (20 °C/min) to 310 °C (5 °C/min, 2 min)	Splitless (260 °C)	0.08–0.15 µg/kg (0.26–0.30 µg/kg)	Low spike (5 µg/kg)=62.81–105.18 Higher spike (10 µg/kg)=68.46–113.86	Sun et al. (2020)
Cachaça	16 US EPA PAHs excluding NPH, ACP, FA	Extraction was done with direct immersion and cooled fiber solid-phase microextraction mode (DI-CF-SPME)	GC-MS Capillary column (30 m × 0.25 mm, 0.25 µm)	70 °C to 120 °C (30 °C/min) to 280 °C (10 °C/min, 10 min)	1. Splitless mode (1 min) 2. Split ratio 1:20 (270 °C)	10–80 ng/L (20–220 ng/L)	74–95	Menezes et al. (2015)
Cachaça	16 US EPA PAHs excluding BbFA	Multiple dispersive liquid-liquid microextraction (MDLME) with chloroform as the extracting solvent	GC-MS; Rtx-5MS (30 m × 0.25 mm, 0.25 µm)	40 °C (2 min) to 100 (12 °C/min) to 300 °C (7 °C/min, 4 min)	(1) Splitless mode (1 min) (2) Split ratio (1:5)	60–1500 ng/L (200–5000 ng/L)	84–116	Will et al. (2018)
Cachaça	15 + 1 EUPAH ^a	Modified QuEChERS method: Extracted using dichloromethane + MgSO ₄ + NaCl and no cleanup steps	GC-MS/MS; DB-EUPAH (30 m × 0.25 mm, 0.25 µm)	40 °C (1.8 min) to 200 °C (70 °C/min) to 240 (6 °C/min) to 280 (10 °C/min) to 310 °C (6 °C/min)	Solvent Vent mode	250 ng/L (1000 ng/L)	84.8–111.5	Da Silva et al. (2019)
Spirits	16 US EPA excluding NPH, ACY, FA and BbFA	Extracted using ultrasound-assisted emulsification microextraction (USAEME)	GC-MS; HP-5MS (30 m × 0.25 mm, 0.25 µm)	75 °C (0.5 min) to 195 °C (15 °C/min, 3.5 min) to 230 (10 °C/min, 8.5 min) to 280 (10 °C/min) to 305 °C (15 °C/min, 6 min)	Splitless (325 °C)	1.8–6.3 ng/L (6–21 ng/L)	84–118	Cacho et al. (2016)
(a) Beer (b) Wine (c) Cider (d) Spirits	16 US EPA PAHs	Concentrated and cleanup using semi-automated SPE [packed with 60 mg of silica-reversed phase with octadecyl functional groups (RP-C18)]	GC-MS; DB-5MS (30 × 0.25 mm × 0.25 mm)	70 °C (2 min) to 240 °C (10 °C/min) to 290 °C (15 °C/min, 12 min)	Splitless (320 °C)	0.02–0.6 ng/L	90–103	Rascón et al. (2019)
Beer	16 US EPA PAHs + ac ^d	Cold fiber solid-phase microextraction (CF-SPME)	GC-MS; HP-5MS (30 m × 0.25 mm, 0.25 µm)	80 °C to 150 °C (30 °C/min) to 210 °C (10 °C/min, 4 min) to 240 °C (15 °C/min) to 280 °C (10 °C/min, 8 min)	Splitless (270 °C)	3–128 ng/L (11–427 ng/L)	80.1–100.3	Resende dos Santos et al. (2021)

N/A Not available

^aBaA, BaP, BbFA, BkFA, BghiP, DahA, CHR, IP, benzo[c]fluorene, cyclopenta[c,d]pyrene, 5-methylchrysene, benzo[j]fluoranthene, dibenzo[a,l]pyrene, dibenzo[a,e]pyrene, dibenzo[a,i]pyrene, and dibenzo[a,h]pyrene^bBenzo[c]fluorene, benzo[c]fluorene, cyclopenta[c,d]pyrene, 5-methylchrysene, benzo[j]fluoranthene, 7,12-dimethyl[a]anthracene, benzo[e]pyrene, 3-methylcholanthrene, dibenzo[a,l]pyrene, dibenzo[a,e]pyrene, dibenzo[a,i]pyrene, and dibenzo[a,h]pyrene^c2-Methyl phenanthrene, 2-methyl anthracene, 9-methyl phenanthrene, 9-methyl anthracene, 2,4-dimethyl phenanthrene, 1,2-dimethyl phenanthrene, 1-methyl pyrene, benzo[e]pyrene, perylene, dibenzo[a,l]pyrene, dibenzo[a,e]pyrene, dibenzo[a,i]pyrene, and dibenzo[a,h]pyrene^d9-Fluorenone, 9,10-anthraquinone, 2-methylanthraquinone, 2-nitrofluorene, 9-nitroanthracene, 3-nitrofluoranthene, 1-nitropyrene, and 5,12-naftacenequinone

Table 3 Prepreparation procedures and the HPLC conditions used for the analysis of PAHs in beverages

PAH	Matrix	Sample preparation	HPLC	Mobile phase	Detector	LOD (LOQ)	Percentage recovery (%)	Article
16 US EPA PAHs excluding ACY	Mate tea	SBSSE with the usage of stir bar coated with PDMS and desorption using acetonitrile/water (4:1)	Vydac™ 201 TP 52 column C18 (250 mm×1.5 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	0.1–8.9 ng/L (0.3–29.7 ng/L)	24–87	Zuin et al. (2005)
16 US EPA PAHs	Tea and coffee	LLE (n-hexane) and C18 cartridge cleanup	RP-18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (75:25 v/v)	Ultraviolet (UV) detector (254 nm)	N/A	68–84	Bishnoi et al. (2005)
16 US EPA PAHs excluding ACP, ACY, DBahA, IP, BghiP	Tea infusions	Headspace SPME with PDMS-DVB fiber	LP-Chrospier PAH RP-18 silica gel (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	4.88–145 ng/L	N/A	Vinas et al. (2007)
16 US EPA PAHs excluding ACY	Tea leaves and tea infusions	Sonication with methanol and in-tube SPME (acetonitrile: water, 5:95 v/v)	Zorbax Eclipse PAH C18 (100 mm×2.1 mm, 3.5 µm) matrix active group	Acetonitrile: water (gradient)	FLD	0.32–4.63 ng/L	70–111	Ishizaki et al. (2010)
US EPA 16 PAHs	Erva mate tea	Extracted using UAE (hexane/acetone) and cleanup using SPE (C ₁₈)	C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water	UV	0.1–0.7 µg/kg (0.5–2.5 µg/kg)	72–100	Vieira et al. (2010)
PHE, FA, Bup	Green tea	SPME's adsorbent produced by immobilization of MWNTs in agarose film	ZORBAX Eclipse Plus C ₁₈ column (100 mm×2.1 mm, 3.5 µm)	Acetonitrile: water (gradient)	UV	0.1–50 ng/L	91.1–107.2	Loh et al. (2013)
US EPA 16 PAHs	Yerba mate tea	UAE (hexane) and cleanup using SPE (silica gel)	C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water	FLD and diode array detector (DAD)	0.01–0.7 µg/kg (0.02–2.5 µg/kg)	89–109	Garcia Londoño et al. (2014)
US EPA 16 PAHs + 2-MN	Dry tea	Extracted using hexane and cleanup with silica gel cartridge	Pinnacle II PAH C18 (150 mm×3 mm, 4 µm)	Acetonitrile: water	FLD	0.01–0.21 µg/kg (0.04–0.71 µg/kg)	> 85	Adisa et al. (2015)
US EPA 16 PAHs	Tea	UAE (hexane) and cleanup with SPE (silica) and elution with hexane	Waters PAH column C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD and DAD	0.01–0.10 µg/kg (0.02–2.5 µg/kg)	> 84.5	Garcia Londoño et al. (2015)
PAH4+PHE, BkFA, DBahA	Tea infusions	MSPE were carried out with Fe ₃ O ₄ @3-(trimethoxysilyl)propyl methacrylate/ionic liquid nanoparticles as the adsorbent	Zorbax Eclipse PAH C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (Gradient)	FLD	0.1–10 ng/L (0.5–34 ng/L)	87.5–104.5	Shi et al. (2016)
PAH8	Tea infusions of yerba mate	Extracted with cyclohexane, dried with anhydrous sodium sulfate, concentrated in rotary evaporator then cleanup with SPE (silica)	Supelcosil™ LC-PAH C18 (250 mm×4.6 mm, 5 mm)	Acetonitrile: water	Spectrofluorimetric	1.2–25 ng/L (4.0–83.5 ng/L)	91–105	Then et al. (2016)
US EPA 16 PAHs excluding ACY	Tea infusions	Saponification (NaOH) followed by LLE (cyclohexane)	Prosphere 300 column C-18 (150 mm×4.6 mm, 5 µm)	Solvent A - water: acetonitrile, Solvent B—acetonitrile: water	FLD	0.2–2.1 ng/L	70–130	Girelli et al. (2017)
PAH4	Dry teas and tea infusions	Modification of QUECHERS: Extracted with ethyl acetate and cleanup using PSA, carbon black, C18 and silica gel sorbents combined	Vydac™ 201 TP 54 C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water	FLD	Tea leaves: 0.2–0.3 µg/kg (0.5 µg/kg) Tea infusions: 0.03–0.05 µg/kg (0.1 µg/kg)	Tea leaves: 67–99 Tea infusions: 54–99	Tifouni et al. (2018)
PAH4	Tea and Tea infusions	Extracted using QUECHERS, cleanup by d-SPE	Hypersil Green PAH Reversed phase (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	Dried Tea: 0.15–0.25 µg/kg (0.5–0.75 µg/kg) Infusions: 30–50 ng/L (100–150 ng/L)	Dried Tea: 51–93 Infusions: 99–107	Zachara et al. (2018)
PAH4+BkFA, DBahA	Tea infusions	DLLME carried out with Ionic liquids (IL) as the extractant	ZORBAX Eclipse Plus PAH Reverse Phase column (150 mm×4.6 mm, 3.5 µm)	Acetonitrile: water (gradient)	FLD	2–30.8 ng/L (6.73–95.2 ng/L)	56–94	Rivera-Yera et al. (2019)
US EPA 16 PAHs + benzo(e)pyrene and excluding ACY	Tea infusions	Extraction done with MASE	Waters PAH C18 column (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (Gradient)	FLD	0.6–28.2 ng/L (1.0–43.8 ng/L)	78–116	Mallana-López et al. (2021)
PAH8 excluding CHR	Instant coffee	Extraction with hexane, cleanup with silica cartridge (hexane as eluting solvent)	Supelcosil LC-PAH C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	0.01–0.05 µg/kg (0.04–0.2 µg/kg)	87–103	Garcia Londoño et al. (2015)

Table 3 (continued)

PAH	Matrix	Sample preparation	HPLC	Mobile phase	Detector	LOD (LOQ)	Percentage recovery (%)	Article
FLR, BaP, BbFA	Coffee brew	Preconcentration of the samples on PS-DVB cartridges and elution with methanol/THF (10:90)	Supelcosil LC-PAH C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	0.76–9.96 ng/L (2.52–33.2 ng/L)	80–106	Houessou et al. (2005)
16 US EPA PAHs excluding NPH, ACP, ACY, FLR, IP	Ground coffee	PLE with hexane/acetone 50/50 (v/v), alkaline saponification, cyclohexane extraction and cleanup with SPE (silica)	Supelcosil LC-PAH C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (Gradient)	FLD	0.11–0.18 µg/kg	64–106	Houessou et al. (2006)
16 US EPA PAHs excluding NPH, ACP, ACY, FLR, IP	Ground Arabica coffee and coffee brew	Coffee samples: PLE with hexane/acetone 50/50 (v/v), then alkaline saponification, cyclohexane extraction and SPE cleanup (silica) Coffee brew: Extracted using alkaline saponification, cyclohexane extraction then cleanup using silica SPE cartridges (silica)	Supelcosil LC-PAH C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (60:40, v/v)	FLD	N/A	N/A	Houessou et al. (2007)
PAH8 excluding IP	Ground coffee	alkaline saponification (KOH), LLE (n-hexane) then SPE (Florisil)	Supelcosil LC-PAH C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	0.016–0.497 µg/kg (0.054–1.666 µg/kg)	68–91	Lee and Shin (2010)
BaA, BbFA, BkFA, BaP	Ground coffee	Extracted using Soxhlet, then dimethylformamide-water (9:1, v/v) and purified by column chromatography on silica gel	Vydac TM 201 TP 54 C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	0.02–0.04 µg/kg	66–87	Tfouni et al. (2012)
PAH4+BaFA excluding CHR	Coffee brew	LLE (cyclohexane) and cleanup using SPE (silica gel)	Vydac 201 TP54 C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	6–10 ng/L	77–87	Tfouni et al. (2013)
16 US EPA PAHs + 1-MN and 2-MN	Ground coffee	Extracted with hexane using a rotor mixer and cleanup with (silica gel)	Phenacel II PAH C18 (150 mm×3 mm, 4 µm)	Acetonitrile: water (gradient)	FLD	0.01–0.21 µg/kg (0.03–0.71 µg/kg)	> 80	Jimenez et al. (2014)
PAH8+PHE excluding BghiP, IP	Coffee brew	MSPE were carried out with Fe ₃ O ₄ @3-(trimethoxysilyl)propyl methacrylate@ionic liquid nanoparticles as the adsorbent	Eclipse PAH C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	0.1–10 ng/L (0.5–34 ng/L)	87.5–104.5	Shi et al. (2016)
PHE, ANT, PYR, BaA, CHR, BkFA, BaP, BghiP	Milk	Alkaline saponification (KOH), LLE (cyclohexane) and cleanup with SPE	Supelcosil LC-PAH C18 (150 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	FLD	LOQ: 24–401.7 ng/L (liquid), 0.0120–0.2008 µg/kg (solid)	89.15–93.78	Naciri et al. (2011)
US EPA 16 PAHs excluding NPH	Milk powders	UAE (hexane) and cleanup with SPE (silica)	C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (gradient)	DAD and FLD	0.0004–9 µg/kg (0.0014–16.4 µg/kg)	> 79	Garcia Londoño et al. (2013)
US EPA 16 PAHs	Milk	LLE (cyclohexane) and filtered with PTFE membrane filters	Phosphor 300 C-18 (150 mm×4.6 mm, 5 µm)	1% acetonitrile in aqueous solution: 99% acetonitrile in aqueous solution	FLD	200–10,000 ng/L (660–33,300 ng/L)	70–115	Girelli et al. (2014)
US EPA 16 PAHs excluding NPH, ACY	Milk	Alkaline saponification (KOH) and cleanup with SPE (C ₁₈)	Envirosep-PP C18 (125 mm×4.6 mm, 5 µm)	Acetonitrile: water (Gradient)	FLD	0.005–0.11 µg/kg	65.2–89.3	Santonicola et al. (2017)
US EPA 16 PAHs excluding ACY	Cachaça	Cleanup with SPE (C18 cartridge) and evaporated to dryness with nitrogen gas	Reverse phase Supelcosil TM LC-PAH C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (Gradient)	FLD	1.08–45.4 ng/L (93–881 ng/L)	81.5–113	Galliano et al. (2007)
BaA+BaFA+BkFA+BaP+DBaA	Cachaça	Extracted with LLE (cyclohexane) then dimethylformamide-water (9:1, v/v) and cleanup on silica gel column	Vydac 201 TP54 C18 (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (Gradient)	FLD	6–90 ng/L	70–96.7	Tfouni et al. (2007)
BaP	Cachaça	Extracted and cleanup using SPE (C18 cartridge) and evaporated to dryness with nitrogen gas	Reversed phase C18 column (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (Gradient)	FLD	30 ng/L (100 ng/L)	82.9–97.0	Caruso and Alaburda (2009)

Table 3 (continued)

PAH	Matrix	Sample preparation	HPLC	Mobile phase	Detector	LOD (LOQ)	Percentage recovery (%)	Article
PAH8+CHR	(a) Red wine (b) White rum	Extraction, concentration and purification were done by using two solid-phase extraction minicolumns in series: (1) octadecylsilica phase (2) silica stationary phase	Supelcosil LC-PAH (250 mm×4.6 mm, 5 µm)	Acetonitrile: water (Gradient)	FLD	0.1–2.0 ng/L (0.2–6.0 ng/L)	(a) 87–100 (b) 88–102	García-Falcón et al. (2005b)

N/A not available

Analysis

To determine the quantity of PAHs in beverages, chromatographic analysis methods using analytical instruments are most commonly employed, such as liquid chromatography (LC) coupled with a fluorescence detector (FLD) (Kayali-Sayadi et al. 2000) and gas chromatography coupled with mass spectrometry (GC–MS) (Purcaro et al. 2007; Schulz et al. 2014; Zhou et al. 2018; Sun et al. 2020).

High-performance liquid chromatography with fluorescence detection (HPLC–FLD) is a highly recommended method for the routine screening purpose of PAHS due to its selectivity and sensitivity. HPLC is also preferable for the quantification of PAH isomers. For instance, the isomers chrysene/triphenylene, the first is a target PAH but the second may interfere with chrysene quantification by gas chromatography (GC) (Zelinkova and Wenzl 2015). Therefore, HPLC should be selected for this analysis as GC is unable to resolve some pairs of peaks.

GC–MS is a widely acclaimed analytical instrument for the analysis of PAHs in food samples, and researchers have been extensively employing GC–MS in their studies. This is because GC has a better resolution efficiency compared to HPLC (Tamakawa 2008) and MS provides high selectivity and sensitivity as well as structural information of PAHs (Plaza-bolaños et al. 2010). GC–MS allows the resolution of PAHs with poor fluorescence, such as NPH, ACY, ACP, and FLR or non-fluorescence PAHs, such as cyclopenta(c,d)pyrene (CPcdP) (Cai et al. 2009). Furthermore, gas chromatography–tandem mass spectrometry (GC–MS/MS) detection is a widely popular method which presents high sensitivity and resolution (Plaza-bolaños et al. 2010).

Based on literature reviews, PAHs in beverages were most frequently analyzed by GC–MS in selected ion monitoring (SIM) mode, where only masses of PAHs of interested are monitored. The limits of detections (LODs), limits of quantifications (LOQs), and percent recoveries for the analysis of beverages using GC–MS and HPLC gathered from different literatures are summarized as shown in Table 2 and Table 3, respectively. For the separation of compounds in complex food samples including beverages, two types of columns that are commonly used in GC analysis are DB-5MS column coated with 5%-phenyl-Arylene-95%-dimethylpolysiloxane (Sadowska-Rociek et al. 2015; Roudbari et al. 2020) and HP-5MS column coated with 5%-phenyl-95%-dimethylpolysiloxane (Aguinaga et al. 2007; Zhou et al. 2018). Whereas for the HPLC analysis, the C18 column or its equivalence, running in isocratic or gradient elution, is favorable for the analysis of PAHs in most of the reported publications as summarized in Table 3 (Garcia Londoño et al. 2015; Shi et al. 2016; Tfouni et al. 2018; Mañana-López et al. 2021). There are also an increasing number of columns which are specifically developed for the analysis PAHs using HPLC

such as Eclipse PAH column, Supelcosil™ LC PAH column, Hypersil Green PAH column, Envirosep-PP, Pinnacle II PAH, Vydac™ and LiChrospher PAH column to name a few (Shi et al. 2016; Thea et al. 2016; Kayali-Sayadi et al. 1999; Santonicola et al. 2017; Adisa et al. 2015; Zuin et al. 2005; Viñas et al. 2007). This indicates the growing importance of the analysis of PAHs.

According to Table 2 and Table 3, it was observed that different results of LODs, LOQs, and recoveries were obtained when different sample extraction methods, cleanup, and instrumental analysis were employed. The lowest LODs obtained using GC–MS for the analysis of tea, tea infusions, ground coffee, coffee brew, milk, and alcoholic beverages were 0.01–0.02 µg/kg, 0.1–0.28 ng/L, 0.03–0.18 µg/kg, 0.67–18 ng/L, 3–1500 ng/L, and 0.02–0.6 ng/L respectively. For HPLC, the lowest LODs reported for tea, tea infusions, ground coffee, coffee brew, milk, and alcoholic beverages were 0.32–4.63 ng/L, 0.2–2.1 ng/L, 0.01–0.21 µg/kg, 0.1–10 ng/L, 0.0004–4.9 µg/kg, and 0.1–2.0 ng/L respectively. Lower LODs in beverages were observed with the usage of HPLC compared to GC. In addition, percentage recoveries were mostly within the range of 70–110% for beverages analyzed using GC and HPLC.

Conclusion

Global concerns have been raised due to the discovery of increasing amounts of PAHs found in beverages, prompting researches to develop sample extraction and analytical methods for the determination of PAHs in the past 16 years. Extraction methods with the usage of green extractants to minimize hazardous organic solvents wastage to the environment are being developed as well as integrated into existing research, and have been used extensively due to their simplicity. Analytical instruments, in particular GC and HPLC with detectors such as MS, MS/MS, FID and FLD are generally favored for the analysis of PAHs; LOD as low as 0.02 ppt level can be accomplished. It was found that QuEChERS is a popular method for the extraction of PAHs from tea, whereas saponification is more commonly used for the extraction of PAHs from both coffee and milk. For alcoholic beverages, LLE, SPE and microextraction techniques are commonly used.

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Declarations

Informed Consent Not applicable.

Conflict of Interest Peng Pau Lian declares that she has no conflict of interest. Lee Hoon Lim declares that she has no conflict of interest.

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