Analytical procedures for determination of phenolics active herbal ingredients in fortified functional foods: an overview

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Abstract
Fortification of foods with phenolic compounds is becoming increasingly popular due to their beneficial physiological effects. The biological activities reported include antioxidant, anticancer, antidiabetic, anti-inflammatory, or neuroprotective effects. However, the analysis of polyphenols in functional food matrices is a difficult task because of the complexity of the matrix. The main challenge is that polyphenols can interact with other food components, such as carbohydrates, proteins, or lipids. The chemical reactions that occur during the baking technologies in the bakery and biscuit industry may also affect the results of measurements. The analysis of polyphenols found in fortified foods can be done by several techniques, such as liquid chromatography (HPLC and UPLC), gas chromatography (GC), or spectrophotometry (TPC, DPPH, FRAP assay etc.). This paper aims to review the available information on analytical methods to fortified foodstuffs while as presenting the advantages and limitations of each technique.

Keywords Polyphenols · Fortified foods · Analytical methods · Medicinal plants

Introduction

In the last decades consumer demands for different food products have changed. Nowadays foods are not intended to only satisfy the hunger for humans but also to prevent nutrition-related diseases or improve the physical and mental wellbeing of consumers [1]. In recent years, functional foods have gained popularity because these products can help reduce the risk of disease. It is estimated by “Market Research” [2] that the global market of the functional food industry will reach $167 billion in 2025. The concept of functional food was first used in Japan in the 1980s. This notion eventually became widespread throughout the world. Although these foods have not yet been defined by legislation in Europe, most agree that functional foods are healthy foods or food components that have a potentially positive effect on health beyond basic nutrition [3].

Polyphenols are secondary metabolites in various plant materials that have many beneficial effects on the human body and health [4]. The phenolic compounds have primarily antioxidant and anti-inflammatory effects; in addition, the results of recent research have shown that they can have a preventive or therapeutic effect on cardiovascular diseases, neurodegenerative disorders, obesity or cancer [5]. For this reason, a number of studies have been published where various foods were fortified with polyphenol-rich plant extract [6–10]. However, the health implications of bioactive polyphenols are determined by their bioavailability to a great extent, which is influenced by many factors, including phenolic structure, chemical interactions, food processing, and matrix. The development of fortified food is valueless if the active ingredients are not stable in the food matrix or if they are not absorbed throughout the digestive system. In this context, encapsulation processes play an instrumental role in protecting the bioactive components in the food matrix as well as favor their absorption in the gastrointestinal tract [11, 12]. Consumers expect that the products they use in everyday life are safe and high quality. Therefore, the development and application of analytical methods in the field of plant-fortified functional food analysis are crucial. However, studies in this direction are considerably complex because herbal preparations contain numerous active ingredients. Moreover, polyphenols may interact with the food matrix components [13, 14]. Spectrophotometric methodologies such as 2,2-diphenyl-1-picrylhydrazyl radical scavenging - (DPPH),...
Folin-Ciocalteu-, or ferric reducing antioxidant power (FRAP) assay are widely used for fortified food analysis [15–17]. In comparison with chromatography-based technologies, these methods are less sensitive and specific. Currently, high-pressure liquid chromatography (HPLC) with diode array (DAD) or mass spectrometry (MS) detection is the most widely used analytical approach for polyphenol analysis in fortified food matrices [18, 19].

This report includes a discussion of how plant extracts appear in fortified functional foods, the effects of the extracts on different foods, the effect of these products on human health, and finally, it describes the analytical methods used for the quantification and determination of phenolic compounds from fortified foodstuffs.

**Application of plant extracts in food**

Over the last decade, foods fortified with herbal extracts rich in phenolic substances have become widespread (Table 1).

As shown in Table 1, plant extracts are typically used in food fortification in the form of freeze-dried. However, phenolic compounds uses are substantially limited due to their instability during food processing and storage or in the gastrointestinal tract. The behavior of phenolic compounds in the human body can be affected by the structure and composition of the food matrix and the class of polyphenol. In liquid matrices, the polyphenols are more readily bioaccessible whereas, if the matrix is solid, the polyphenols contained must first be extracted to be bioaccessible and potentially bioavailable. In the case of the human body, the extraction is carried out by the gastrointestinal tract where both the mechanical action during mastication and the chemical action during the digestive phase contribute to the extraction of phenolics from solid matrices. The extraction of polyphenols from solid matrices is influenced by various factors such as temperature, pH, the type of solvent used, and so on. These variables can also affect the extraction efficiency of different polyphenols in the gastrointestinal tract [33]. For example, studying the bioaccessibility of olive polyphenols (verbascoside, hydroxytyrosol, and oleuropein) in fortified taralli, Cedola et al. [26] realized that the combined effect of enzymatic activity and pH changes helps degrade part of the bioactive compounds in the gastric and intestinal phase. Polyphenols have been shown that can reduce the digestive rate of starch, thus modulating the glycemic response to carbohydrates [34]. Consequently, extracts from plants have recently been incorporated in cereal-based products to help alleviate type 2 diabetes mellitus [35]. However, some studies suggest that polyphenols have lower effects on starch digestion in the case of fortified food. For example, Kan et al. [36] observed lower starch digestion inhibition when bread was fortified with berry extracts when compared to the co-ingestion of berry polyphenols with bread during in vitro assay. Coe and Ryan [37] applied an in vitro dose–response analysis to determine the optimal dose of a baobab fruit extract and green tea extract for reducing rapidly digestible starch in white bread. Although bread fortified with tea extract (0.4%) and baobab fruit extract (1.9%) did not reduce the satiety or glycemic response, white bread with added baobab fruit extract increased insulin economy by reducing the amount of insulin needed for given blood glucose. In some cases, lipids can also have a positive effect on the bioavailability of polyphenols, as they are able to "capture" and protect them from degradation in the gastrointestinal tract or the formation of insoluble complexes [38]. In a previous study, Ortega et al. [39] reported that higher fat content might have a positive effect on the stability of cocoa polyphenols, possibly due to the improved micellarization during digestion. Nowadays, dairy products are one of the most ideal carrier matrices for the delivery of bioactive plant ingredients to the human body. For example, yogurt is an excellent delivery vehicle for phenolic compounds of plant extracts because the low pH (~4.1–4.5) of yogurt contributes to the stability of phenolic compounds during storage [40], while the presence of proteins maintains the integrity of phenolic compounds during digestion, increasing their bioaccessibility [41]. Other dairy products, such as cheese or milk can also serve as a suitable matrix for the controlled release of phenolic compounds. Lamothe et al. [42] showed that the green tea extract addition to cheese and milk promoted polyphenol-protein complex formation, which significantly improved polyphenol stability in a simulated gastrointestinal environment and enhanced the antioxidant activity.

Polyphenols in free form may also have negative effects on the taste of different food due to their strong astrin- 
gency effect [6]. Moreover, freeze-dried plant extracts with larger particles (> 25 micron) often cause a sandy mouthfeel [43]. To avoid these drawbacks, delivery systems have been developed, and among them, encapsulation plays a predominant role. Encapsulation is a process to entrap phenolic active ingredients within a wall material. The wall materials used for encapsulates must be food-grade, biodegradable, and stable in the food system during processing, storage, and consumption. Among the used wall materials, polysaccharide- and protein-based polymers are widely used for encapsulation. According to the obtained particle size, capsules are called micro- or nanocapsule. Microparticles are generally in the 1–123 µm size range, while the size of nanoparticles ranges approximately from 1 to 200 nm. Commonly applied encapsulation techniques for the purpose of encapsulating phenolic extracts are as follows: anti-solvent precipitation, electrospraying, spray-drying, and atomization/coagulation (Table 2).
## Table 1  Applications of plant extracts in various foodstuffs

| Plant extract                        | Applied form | Concentration of plant extract used | Food product tested | Main results                                                                                                                                                                                                 | References |
|--------------------------------------|--------------|-------------------------------------|---------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------|
| **Dairy products**                   |              |                                     |                     |                                                                                                                                                                                                             |            |
| Roselle calyces (*Hibiscus sabdariffa* L.) | Freeze-dried | 1%                                  | Goat-milk yogurt    | Increased α-glucosidase inhibitory activity and decreased pH. The fortified yogurt stored under refrigerated conditions (4 °C) had acceptable shelf life during the study period (15 days)                                    | [20]       |
| Safflower (*Carthamus tinctorius* L.) | Freeze-dried | 0.1, 0.5, and 1.0%                  | Yogurt              | Increased protein, fat, lactose, TPC, TFC, and antioxidant activity. Lower moisture content and pH value. Reduced ROS production in LPS-induced HT-29-cells. Safflower extract downregulated α-glucosidase and lipase activities | [21]       |
| Argel (*Solenostemma argel* Hayne)   | Freeze-dried | 0.1 and 0.2 g/100 mL                | Set-type yogurt     | Increased acidity, water-holding capacity, viscosity, TPC, and antioxidant activity. Reduced pH, syneresis, texture, and TBARS values. Also reduced L* value and increased a* and b* values                                           | [22]       |
| British yellowhead (*Inula britannica* L.) | Freeze-dried | 0.25, 0.5, 0.75, and 1%              | Cheddar type cheese | Higher extract concentrations resulted in higher protein and ash contents, with a concomitant decrease in pH, total solid, and fat content. Higher TPC and antioxidant activities compared to the control. Fortification gave a more desirable texture without change of taste and odor of the cheeses | [23]       |
| **Cereal-based products**            |              |                                     |                     |                                                                                                                                                                                                             |            |
| Blue pea (*Clitoria ternatea* L.)    | –            | 5, 10, 15, and 20%                  | Sponga cakes        | Higher TPC and antioxidant activity. Reduced TBARS values. Lower L*, a*, and b* values were observed with increasing concentration of blue pea                                                                | [24]       |
| Green tea (*Camellia sinensis* L.)   | Freeze-dried | 0.5, 0.8, and 1.1%                  | Rye breads          | Catechins in rye bread samples significantly increased with the increasing level of green tea extract. Catechin stability was in the order: epigallatocatechin < epigallatocatechin gallate < epicatechin gallate. The antioxidant effectiveness decreased in the following order: 1.1% > 0.8% > 0.5% > control | [25]       |
| Olive (*Olea europaea* L.)           | Liquid       | 75 g                                | Taralli             | Higher TPC, TFC, and antioxidant activity compared to the control. Decreased abundant polyphenol present in taralli in the gastric and intestinal phase. Polyphenol stability during in vitro digestion was in the order: verbascoside > hydroxytyrosol > oleuropein | [26]       |
| Plant extract          | Applied form            | Concentration of plant extract used | Food product tested       | Main results                                                                 | References |
|------------------------|-------------------------|------------------------------------|---------------------------|------------------------------------------------------------------------------|------------|
| Glasswort (*Salicornia europaea* L.) | vacuum oven-Dried | 20 mL/50 g                        | Durum wheat fresh pasta   | Increased antioxidant activity compared to the control. Higher TPC and TFC contents after digestion | [27]       |
| Spearmint (*Mentha spicata* L.)       | Liquid                  | 2.5, 5, and 7.5%                  | Bread                     | Higher TPC, DDPH radical scavenging activity, and FIC ability compared to control. Spearmint extract at 2.5% showed the highest organoleptic properties | [28]       |
| Turmeric (*Curcuma longa* L.)         | –                       | 250, 500, and 750 mg/kg           | Fresh lamb sausages       | Reduced lipid oxidation and increased antioxidant capacity                    | [9]        |
| Perilla (*Perilla frutescens* L.)     | Freeze-dried            | 0.03%                             | Surimi fish balls         | Retarded lipid and protein oxidation. Reduced TBARS values and protein carbonyl contents | [29]       |
| Drumstick-tree (*Moringa oleifera* L.) | Powdered               | 0.1%                              | Goat meat patties         | Higher TPC compared to control. Retarded lipid peroxidation                    | [30]       |
| Cinnamon (*Cinnamomum zeylanicum*)    | Powdered                | 0.5 and 1%                        | Dark chocolate            | Higher TPC and decreased texture properties (hardness) as compared to the control. A whitening of chocolate surface was observed during storage (60 days) | [31]       |
| Roselle calyces (*Hibiscus sabdariffa* L.) | Suspension             | 20.0%                             | Cupcakes                  | Fortification increased the, ash, fiber, ascorbic acid, and total anthocyanins content in the cupcakes. Also increased a* value and decreased b* and L* values | [32]       |
| Nettle (*Urtica dioica* L.)           | Freeze-dried or syrup   | 2.0%                              | Milk, semisweet, and dark chocolates | Higher TPC, chlorogenic acid and flavonoid derivate compared to control. Nettle extract increased the astringency and bitterness. Dark chocolates showed better sensory properties than semisweet and milk chocolates | [6]        |

*TPC* total phenolic content, *TFC* total flavonoid content, *ROS* reactive oxygen species, *LPS* lipopolysaccharide, *HT-29* the human colorectal cell line, *TBARS* thiobarbituric acid reactive substances, *DDPH* 2,2-Diphenyl-2-picrylhydrazyl, *FIC* ferrous-ion chelating.
Table 2  Studies on encapsulation of polyphenols for food fortification

| Food product tested | Plant extract            | Wall material          | Encapsulation method             | Capsule properties                                                                 | Main results                                                                                                                                                                                                 | References |
|---------------------|--------------------------|------------------------|----------------------------------|------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------|
| White and milk chocolate | Cinnamon (Cinnamomum burmannii Blume) | Shellac and xanthan gum | Anti-Solvent precipitation       | The average size of the colloidal nanoparticles was 191 nm, while the size of the lyophilized nanoparticles ranges from 9 to 104 µm | Higher TPC and antioxidant activity of chocolates. The presence of the particles up to 2% does not alter the fineness of the chocolate. Slower polyphenol release | [44]       |
| Biscuits            | Green tea (Camellia sinensis L.) | Gelatin and zein       | Electrospraying                  | Gelatin particles were quasispherical. Zein particles were smaller, had a rougher surface and a greater content of small fibrils. In both cases, particles had a size in the submicron range | Encapsulation efficiency of catechins was close to 90 g/100 g (gelatin) and 85 g/100 g (zein). Decreased total catechin recovery from biscuits enriched with microcapsule | [45]       |
| Cottage cheese      | Rosemary (Rosmarinus officinalis L.) | Alginate               | Atomization/coagulation          | Round or pear-like form. Small brown droplets were also noticed, which was related to the presence of the extract. Particle size ranges between 51.1 and 122.6 µm | Encapsulation efficiency of around 100%. Unchanged nutritional values compare to the control. Micronencapsulated extracts showed better antioxidant properties throughout storage | [12]       |
| Yogurt              | Doum tree (Hyphaene thebaica)    | Lecithin and chitosan  | Liposomes                         | The sizes of liposomes were ranges between 261 and 483 nm depending on the concentration of doum extract | Encapsulation efficiency was higher than 70%. 5% liposome addition was recommended to increase the antioxidant activity of yogurt | [46]       |
| Milk                | Turmeric (Curcuma longa L.)      | Dextrin                | Spray-drying                      | The mean diameter of turmeric extract nanoemulsions powder was approximately 200 nm | Nanoemulsions effectively protected the encapsulated turmeric extract from degradation during storage. Milk showed noticeable yellowing, particularly for higher concentrations (> 10 mg/mL). Encapsulation was able to protect curcuminoids during digestion | [11]       |

TPC total phenolic content
Sampling, clean-up, and extraction techniques

Correct sample preparation is an important step for the success of analytical processes. Although the interactions between polyphenols and macronutrient of foods generally have a positive effect on the bioaccessibility of phenolics [38, 42], they can cause a multitude of problems in the analysis, including the generation of emulsions, sample turbidity, ion suppression in MS detection, blockage, or irreversible damage/adsorption onto stationary phases of HPLC, etc. [47, 48].

Functional foods fortified with plant extracts can be classified into three broad categories according to their rheological properties: liquid (milk), semi-solid (yogurt, mayonnaise), and solid (chocolate, cake, cheese, etc.) samples. In general, solid samples require more complex and time-consuming treatments than liquids or semi-solid samples. These steps mainly depend on the food matrix, the plant extracts, most specifically the chemical properties of active ingredients, and the type of used analytical techniques. The main challenge for analysts is to maximize recovery of the analytes and minimize the interferences by use of appropriate extraction and clean-up treatments [49]. The general scheme of pretreatment and extraction methods of polyphenols from fortified foods is shown in Fig. 1.

Solid samples are usually subjected to particle size reduction either by grating [50], crushing [51], or grinding [52]. It is well known that surface area increases with the decrease in particle size. The main advantage of this step is increasing the interaction with solvent and mass transfer of phenolics. For dairy products, while some authors measure liquid and semi-liquid samples immediately after centrifugation and filtration [28], others use lyophilization as a sample preparation step and then use the powdered sample for further clean-up stage [53] or extraction [12]. To eliminate the water content, fortified cereal-based and meat products, such as pasta [27], bread [25], cookies [54], or sausage [9], are also often lyophilized. This step is mainly necessary for high-fat content samples before lipid removal because many organic solvents cannot easily penetrate food containing much water, and therefore degreasing would be inefficient. Solvent extraction (SE) using a vortex mixer is one of the most commonly used and easiest methods for removing the lipid phase from food matrixes. This method generally includes initial extraction with n-hexane (usually three times), centrifugation and/or filtration, discarding of fat-containing supernatants, and removal of residual organic solvent from the defatted sample by air-drying for 24 h [55]. Its main drawbacks are the time and effort required to carry out the extraction and the large volumes of applied organic solvents [56]. Furthermore, hexane as a lipophilic solvent could extract lipophilic phenolic compounds alongside...
targeted lipids [57]. Martini et al. [58] recently reported a non-solvent method. The process is based on centrifugation and allows the cocoa butter to precipitate in 10 min from the chocolate extract.

In most cases, especially for dairy products, another main step in sample preparation is the precipitation of contaminant protein because the interaction between polyphenols and proteins can affect the release of phenolics from the matrix during the extraction step [49]. Proteins in foods are usually removed using isoelectric precipitation by a mineral acid such as hydrochloric acid and sulfuric acid at its isoelectric point during the pretreatment of samples. Despite this, Zhang et al. [59] in their study indicated that a saturated solution of lead acetate and 5% potassium oxalate is more suitable for protein removal from fortified milk than isoelectric precipitation. Acid precipitation combined with alkaline treatment was used for purslane-fortified Greek-style yogurt by El-Sayed et al. [17]. For this, yogurt samples were homogenized with distilled water, and the pH was readjusted by the addition of 2.5 mL HCl to 4. After centrifugation, the pH of the supernatants was adjusted to pH 7.0 using NaOH and re-centrifuged for the further precipitation of proteins.

For fortified foodstuffs, there can be no specific extraction rules. The main considerations that the analyst must be considered for the extraction, are the nature of the food and the nature of the ingredients used for fortification [60]. The techniques range from conventional solvent extraction (maceration with shaking/stirring) [7, 25, 40] to modern methods like ultrasound-assisted extraction [19, 61, 62] and microwave-assisted extraction [63]. In recent years, these methods have been discussed by many authors in several excellent review articles [64–67]. Therefore, this part of the review focuses on a brief presentation of the extraction conditions used to extract phenolic compounds from various fortified food matrices (Table 3).

In some cases, solid-phase extraction (SPE) has also been reported as a method for purifying phenolic compounds from fortified liquid samples before instrumental detection. For instance, SPE on a column of modified silica (C18) allowed the recovery of phenolics by elution with methanol [69] or N, N-Dimethylformamide [59] from fortified milk beverage.

### Analytical methods
#### Spectrophotometric methodologies

The indirect measurement of phytochemicals has been applied for decades. The used spectrophotometric techniques are based on electron/proton transfer reactions and usually are linked to the antioxidant capacity. Depending on what kind of reaction involved, the assays can be classified into two groups: electron transfer (e.g. DPPH, ABTS, FRAP, TPC) and hydrogen atom transfer-based methods (e.g. ORAC). It is important to note that no single method is enough to determine the antioxidant property since different assays can give widely different results. Differences between methods can be ascribed to reaction media. Certain phenolic compounds may not be soluble in reaction media cannot express radical scavenging activities. For example, some assays measure only the hydrophilic antioxidants (e.g., Folin, FRAP, or ABTS), while others (e.g., DPPH) detect only those soluble in alcoholic solvents [70]. In addition, it should be noted that non-phenolic components found in the food matrix may also cause interference during antioxidant analysis. The Folin-Ciocalteu assay is the most widely used procedure for quantification of total phenolics in plant extract supplemented food. This assay is a colorimetric method based on electron transfer reactions between the Folin-Ciocalteu reagent and phenolic compounds. However, the method is not specific for total phenolic content determinations, as the Folin-Ciocalteu reagent can react with food compounds (especially if they are present in large amounts), such as vitamins, amino acids, proteins, carbohydrates, organic acids, and so on, thereby skewing the results. For example, sugars in high-sugar foods (e.g., chocolate) can cause interference if present in high concentrations. Barišić et al. [71], in their study, compared the classical and the modified Folin-Ciocalteu assay (acidic

### Table 3 A brief summary of the experimental conditions for conventional and nonconventional extraction techniques for fortified food material

| Extraction methods | Maceration with shaking | Ultrasound-assisted extraction | Microwave-assisted extraction |
|--------------------|-------------------------|-------------------------------|-------------------------------|
| **Matrix**         | Yogurt, cheese, cupcake, bread | White chocolate, soy milk, durum wheat spaghetti, biscuit | Dark chocolate |
| Common solvents used | Methanol, water, aqueous ethanol/methanol, or acidified methanol | Methanol, aqueous methanol, or acidified acetone/ethanol | Aqueous methanol |
| Temperature (°C)   | Ambient or can be heated | Ambient or can be heated | 60 |
| Time required (min) | 45–300 | 15–60 | 5 |
| Sample: solvent ratio (g/mL) | from 1:1 to 1:40 | from 1:4 to 1:20 | 1:20 |
| Reference | [7, 28, 40, 68] | [10, 19, 52, 61] | [63] |
and on the other hand, that higher temperatures (> 110 °C) known that the stability of phenolic compounds differs [74], the type of phenolic compounds. On one hand, it is well extracts, which was explained by the baking process and their study showed a different phenolic distribution between cal reactions that occur during the baking process may also affect the results of measurements. Baiano et al. [73], in another study, Jahangir et al. [31] measured 9.86 mg GAE/g of total phenolic value for dark chocolate. In both studies, the standard Folin-Ciocalteu assay was used under non-acidic conditions and the results obtained were consistent with the values measured by Barisić et al. [71], which ranged from 1.70 to 3.63 mg GAE/g for milk and 7.54–12.71 mg GAE/g for dark chocolate, confirming the fact that these results are often overestimated due to sugar interference. Similar observations were also made for yogurts. However, the Folin-Ciocalteu reactivity of yogurts is derived from the degradation of milk protein, which may result in the release of phenolic amino acids and non-phenolic compounds such as sugars and proteins, which can interfere with the analysis [28]. For example, Kim et al. [40] observed an increasing tendency in total phenol content during storage (7 days) of yogurt in both the presence and absence of lotus leaf, which was explained by proteolysis of milk protein, which released amino acids with phenolic side chains. Besides, the authors noted that the metabolism of microbes could also have produced new phenolic acids, which could contribute to the increased total polyphenol values. In contrast, Cho et al. [72] reported a temporary decrease in the total phenolic value of plain and olive leaf supplemented yogurt due to the decomposition of polymeric phenolic compounds in the presence of lactic acid bacteria during refrigerated storage (15 days).

In the case of bread, it has been shown that the chemical reactions that occur during the baking process may also affect the results of measurements. Baiano et al. [73], in their study showed a different phenolic distribution between the crust and crumb bread enriched with vegetable waste extracts, which was explained by the baking process and the type of phenolic compounds. On one hand, it is well known that the stability of phenolic compounds differs [74], and on the other hand, that higher temperatures (> 110 °C) enhance the Maillard reaction [75]. Since the temperature of the crumb never exceeds 100 °C while the crust can reach also higher temperatures than 205 °C, it can be assumed that in the work of Baiano et al. [73], the heating altered the phenolics to different extents and in different ways in the inner and outer part of the loaf.

Another most frequently employed spectrophotometry method is the DPPH (2,2-diphenyl-1-picrylhydrazyl) radical scavenging assay. It is an accepted method for screening the antioxidant activity of plant extracts and based on the electron donation of antioxidants to neutralize DPPH radical. However, the application of this assay is limited by certain drawbacks, similar to the Folin-Ciocalteu assay. Feng et al. [76], in their study, mentioned that yogurt itself has certain antioxidant effects as a result of a lot of amino acids and small molecular peptides with antioxidant activity produced during its fermentation. Fadavi and Beglaryan [14] reported that the whey proteins containing active groups can reduce the antioxidant activity of peppermint-enriched UF-Feta cheese. Besides, it was also shown that with an increase in rennet values, antioxidant activity decreases due to the interaction of intermediate size peptides of cheese with polyphenols of peppermint. A similar observation was reported in the ferric reducing antioxidant power (FRAP) assay. For example, El-Sayed et al. [17] described a reduced antioxidant activity of purslane-fortified Greek-Style yogurt during cold storage due to milk protein–polyphenol interactions. Another major limitation of the DPPH assay is the overlapped spectra of compounds that absorb in the same wavelength range as DPPH. For example, Raikos et al. [77] attributed the decreased antioxidant activities of salal berry-fortified yogurt beverages to the loss of fruit anthocyanin content. In another study, Shori et al. [28] found a weak correlation between total phenolic content and DPPH radical scavenging activity of spearmint-fortified bread, thereby pointed out that other constituents, such as proteins and amino acids, may also have affected the scavenging activity. Furthermore, Bhat et al [78] observed that DPPH scavenging activities of bakery products are also influenced by the Maillard reaction products (e.g. melanoïdins).

High performance liquid chromatography

In comparison to spectrophotometric chromatography, high-performance liquid chromatography (HPLC) allows more accurate determination for polyphenols. HPLC (or, more recently ultra-high-performance liquid chromatography; UHPLC) with UV–Vis spectroscopy and/or mass spectrometry (MS) is one of the most common approaches for quantitative analysis of phenolic compounds in fortified food samples. Generally, the MS system is coupled with low-resolution mass analyzers (LRMS), especially triple quadrupole (TQ) mass spectrometers. For example, 8 polyphenol compounds were quantified by UHPLC-TQ MS/MS in sausages fortified with grape seed extract [13]. Oh et al. [79] quantified 10 polyphenols in yogurt fortified with Morus alba leaf extract using TQ. In another study [80], 13 polyphenols were quantified by LC-TQ MS/MS in milk fortified with pomegranate peel extract. High-resolution mass spectrometry (HRMS) has become increasingly prominent in recent years for the determination of polyphenols in food. In comparison to LRMS techniques, the HRMS becomes highly advantageous when working with complex matrices, which main
lead to incorrect determinations and even false positive or negative results. Thus, every HPLC methodology should be validated to evaluate the ability of the method to provide reliable quantitative results. Zhang et al. [59] validated a method for the determination of four flavone C-glucosides (homoorientin, orientin, vitexin, and isolvetin) in bamboo leaves-fortified milk by RP-HPLC–DAD wherein several performance characteristics such as linearity, repeatability, recovery, LOD, and LOQ were evaluated. A linear calibration curve was obtained with $r^2 > 0.9995$. The stability of the method was examined by estimating the precision in terms of repeatability and was found to be acceptable with values of $\leq 3.2\%$ for all compounds. Recoveries were in the range 81–92%. The LOD and LOQ values were less than 0.03 and 0.09 µg/mL, respectively. Moreover, there were no impurities or co-elution observed (match factor $\geq 98\%$). A method was developed and validated for the simultaneous quantification of flavan-3-ols, glycosylated flavonols, and benzoic acid derivatives in sausages fortified with grape seed extracts by Ribas-Agustí et al. [13]. The method was performed with a LOD ranging from 1 to 60 mg/100 g depending on the phenolic compounds. In this study, the recovery of procyanidins and epigallocatechin gallate ranged from 61 to 69%, which can be explained by the fact that these compounds may interact with the proteins of sausages. With other phenolic compounds, the recoveries changed between 75 and 96%. Wang and Zhou [89] described an HPLC–PDA method to determine the catechins in bread fortified with green tea extracts. The calibration curves for epicatechin, epigallocatechin, epicatechin gallate, epigallocatechin gallate, catechin gallate, and galloocatechin gallate were linear between 1 and 50 ppm, respectively. The determination coefficients were $\geq 0.999$; the recovery rate varied from 92 to 94%. The authors also showed that the retention levels of green tea catechins in freshly baked bread were ca. 83% and 91%. Overall, one piece of bread (53 g) contained 150 mg of green tea extract/100 g of flour provided 28 mg of tea catechins, which was 35% of those infused from one green tea bag (2 g). The losses were be explained by the isomerization/epimerization and degradation of tea catechins during the various bread-making stages including mixing, thawing, proofing, and baking.

**Gas chromatography**

Few studies have used gas chromatography for the analysis of polyphenols in fortified foods, because these compounds are not volatile. Therefore, before the injection onto GC, a derivatization step is required to ensure good vaporization of the sample and obtain volatile and thermostable derivatives [90]. Derivatization of phenolic compounds in fortified foods can be performed at 70°C in 20–30 min. Trialkylation is the preferred derivatization method to increase the volatility of phenolic compounds. Although there exists...
| Plant extract                | Analyte                                      | Food product analyzed | Measurement method                     | Stationary phase | Mobile phase composition and elution program                                                                 | References |
|-----------------------------|----------------------------------------------|-----------------------|----------------------------------------|------------------|----------------------------------------------------------------------------------------------------------------|------------|
| Lemon balm (Melissa officinalis L.) | Rosmarinic acid                             | Cupcake               | UPLC-DAD-ESI(−)-MS, λ = 280 and 370 nm | Spherisorb S3 ODS-2 C18 (4.6×150 mm; 3 μm) 0.5 mL/min, 35 °C | 0.1% formic acid in H₂O (A), 100% ACN (B), 5 min: 15%B, 5–10 min: 15–20%B, 10–20 min: 20–25%B, 20–30 min: 25–35%B, 30–40 min: 35–50%B | [7]        |
| Olive (Olea europaea L.)    | Verbascoside, hydroxytyrosol, oleuropein    | Taralli               | HPLC-DAD, λ = 240, 280, and 330 nm     | Zorbax C18 (150×4.6 mm; 5 μm) 1 mL/min, n.r | 2.5% acetic acid in H₂O (A), 100% ACN (B), 0–10 min: 10–20%B, 10–35 min: 20–40%B, 35–45 min: 100%B, 45–50 min: 100–10%B | [26]       |
| Chinese cinnamon (Cinnamomum cassia) | Coumaric acid, syringic acid, ferulic acid, quercetin, quercetin-3-rhamnoside, kaempferol, cinnamaldehyde | Yogurt                | HPLC–UV, λ = 270 and 360 nm            | Ascentis C18 (250×4.6 mm; 5 μm) 1 mL/min, n.r | 0.1% formic acid in H₂O (A), 100% ACN (B), 0–0.5 min: 0–3%B, 0.5–10 min: 3–10%B, 10–34 min: 10–40%B, 34–40 min: 40–100%B | [41]       |
| Lemon balm (Melissa officinalis L.), peppermint (Mentha piperita L.), lavender (Lavandula angustifolia L.) rosemary (Rosmarinus officinalis L.), sage (Salvia officinalis L.) | Rosmarinic acid, chlorogenic acid, caffeic acid, luteolin | Goat's milk | HPLC-DAD, λ = 278 nm | Zorbax C18 (250×4.6 mm; 5 μm) 1 mL/min, n.r | 2% formic acid in H₂O (A), 2% formic acid in ACN (B), 0–0.25 min: 90–60%A, 2.5–50 min: 60–30%A | [18]       |
| Korintje cinnamon (Cinnamomum burmannii Blume) | Catechin, epicatechin, procyanidin B1 and B2, 3,4- dihydroxybenzaldehyde, p- hydroxybenzaldehyde, protocatechuic acid, apigenin | White chocolate       | UPLC-ESI(+)−Q-TOF-MS                  | Acquity HSS T3 (100×2.1 mm; 1.8 μm) 0.4 mL/min, 40 °C | 0.1% formic acid in H₂O (A), 0.1% formic acid in ACN (B), 0–1 min: 3%B, 7 min: 15%B, 14 min: 22%B, 17 min: 30%, 22–24 min: 100%B, 26–30 min: 3%B | [19]       |
| Sakura green tea (Camellia sinensis L.), turmeric (Curcuma longa L.) | Hydroxycinnamic acids, hydroxybenzoic acids, flavan-3-ols, ellagitannins, flavonols, flavones | Dark chocolate        | LC-ESI(−)-Q-TOF-MS/MS                 | HSSil C18 (250×4.6 mm, 5 μm) 1.0 mL/min, n.r | 1% formic acid in H₂O (A), 1% formic acid in ACN (B), 0–0.5 min: 4%B, 0.5–60 min: 4–30%B, 60–66 min: 100%B | [58]       |
| Plant extract       | Analyte                                                                 | Food product analyzed | Measurement method                          | Stationary phase | Mobile phase composition and elution program                  | References |
|---------------------|--------------------------------------------------------------------------|-----------------------|---------------------------------------------|------------------|--------------------------------------------------------------|------------|
| Olive (*Olea europaea* L.) | Oleuropein                                                               | Yogurt                | HPLC-DAD, $\lambda = 280$ nm                | Supelcosil LC-ABZ | 1% formic acid in H$_2$O (A), 100% MeOH (B), 0-3 min: 5–13%B, 3-13 min: 15–25%B, 13-25 min: 25–35%B, 25-35 min: 35–45%B, 35–40 min: 45–50%B, 40-45 min: 50–100%B, 45–50 min: 100–5%B [72] |
| Nettle (*Urtica dioica* L.) | Hydroxycinnamic acids, hydroxybenzoic acids, flavan-3-ols, glycosylated flavonoids | Bread                 | UHPLC-DAD-ESI(−)-MS/MS, $\lambda = 254$ and 280 nm | Syncronis C18 column | 0.01% acetic acid in H$_2$O (A), 100% ACN (B), 2 min: 5%B, 2–12 min: 5–95%B, 12–20 min: 95–5%B [51] |
| Burnet rose (*Rosa spinosissima* L.) | Flavonols, flavan-3-ols, anthocyanins, ellagitannins                    | Yogurt                | HPLC-PDA, $\lambda = 254$, 280, 360, and 520 nm | Cadenza Imtakt CD-C18 | 4.5% formic acid in H$_2$O (A), 100% ACN (B), 0–1 min: 5%B in A, 1–20 min: 25%B in A, 20-26 min: 100%B, 26–27 min: 5%B in A [85] |
| Cistus (*Cistus incanus* L.) | $p$-coumaric acid, ferulic acid, rutin, ellagic acid, phloridzin, 3-hydroxybenzoic acid, (+)-catechin, protocatechuic acid, gallic acid | Wheat bread            | HPLC-DAD, $\lambda = 320$ and 280 nm         | Cosmosil SC18-MS-II | 2% acetic acid in H$_2$O (A), 100% MeOH (B), 0 min: 95%A, 10 min: 70%A, 25 min: 50%A, 35 min: 30%A, 40 min: 95%A [86] |
| Rosemary (*Rosmarinus officinalis* L.) | Rosmarinic acid                                                         | Jelly candy           | HPLC-DAD, $\lambda = 280$ and 330 nm         | Zorbax SB-C18 | 0.05% formic acid in H$_2$O (A), 100% ACN (B), 0 min: 5%B, 10 min: 15%B, 30 min: 25%B, 35 min: 30% BA, 50 min: 55% BA, 55 min: 90%B, 70 min: 100%B [87] |
| Green tea (*Camellia sinensis* L.) | (−)-epigallocatechin gallate, (−)-epicatechin gallate, (−)-epigallocatechin, (−)-epicatechin, (−)-gallo| Biscuits              | HPLC-DAD, $\lambda = 280$ nm                 | Zorbax SBC8 (150 × 4.6 mm; 3.5 µm) | 100% ACN (A), 0.1% H$_3$PO$_4$ in H$_2$O, started with 100 mL/100 mL of eluent B, decreasing to 90 mL/100 mL B in 20 min and to 85 mL/100 mL in 60 min [45] |
a great variety of commercially available silylating reagent, the most common in the literature surveyed regarding phenolic compounds has been bis(trimethylsilyl)trifluoroacetamide (BSTFA). Using an appropriate polar solvent such as pyridine can also favor the dissolution of the analyzed material in the derivatizing reagent. The reported works (only two studies) were using FID [91] and MS [92] detection for the analysis of fortified yogurts and edible oils, respectively. Fused silica capillary columns with lengths of 30 m and inner dimensions of 0.25 mm were used in both reported studies. The used column coating material was 5% phenyl-95% dimethyl-polysiloxane (HP-5 and ZB-5). The temperature program is generally based on gradients, using initial temperatures ranging from 70 °C to 80 °C, and final temperatures between 300 °C and 320 °C, achieved in different steps and with rate increases ranging from 4 to 20 °C/min. The authors have injected 1 µl derivatized sample volume into the columns at a split (1:20 ratio) or splitless mode. Helium is used as the carrier gas at a flow-rate of between 0.6 and 2.4 ml/min.

Karaaslan et al. [91] have identified nine individual bioactive phenolic compounds in callus yogurt by GC-FID. According to the data, approximately 72% of the polyphenols present in the yogurt were identified, and the total amount of characterized and quantified phenolic compounds from callus extract-fortified yogurt was 55.8 mg/L. Although these data indicate that the used method was acceptable for the phenolic analysis in fortified yogurt samples, the authors suggest a stronger separation system such as GC–MS/MS or LC–MS for better detection of phenolic compounds found in foods. In another study, Salta et al. [92] quantified 17 phenolic compounds in enriched vegetable oils (olive oil, sunflower oil, palm oil, and a vegetable shortening) by GC–MS analysis. After the olive leaf extract enrichment, oleuropein was predominated in all cases. Moreover, supplementation of olive oil with the extract resulted in a concentration increase of tyrosol, hydroxytyrosol, maslinic acid, caffeic acid, quercetin, protocatechuic acid, and vanillethanediol. Besides, the authors reported good linearity in the range of quantitation limit and up to 20-fold concentration for each phenolic compound.

**Conclusion and future perspectives**

As food fortification with plant extracts is becoming more popular around the world, there will be an increasing need to monitor the quality and safety of these products. However, the analysis of polyphenols is relatively difficult because of the complexity of food matrices. In this study, we pointed out that, besides the interactions between polyphenols and food ingredients, new compounds that formed during the technological process may affect the results of
measurements. From the literature, it is clear that the utilization of encapsulated polyphenols could effectively improve the performance of functional foods.

Although most studies on the analytical characterization of plant-fortified foods use classical pretreatment and extraction techniques for sample preparation, recent sample preparation trends follow an even more straightforward and economic analysis. During the past decade, several new analytical approaches have been appeared and used for the polyphenol analysis from different non-fortified food. An example is the Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) technique, which was successful adopted for the determination of polyphenolic compounds from non-fortified solid [93] and semi-solid [94] as well as liquid [95] samples.

Based on the available literature, it could be stated that the use of spectroscopic techniques is limited by the lack of their specificity. All in all, rapid, accurate, and sensitive chromatographic techniques are becoming increasingly important for polyphenol analysis. Taking into account the complexity of fortified foodstuffs, the use of LRMS and HRMS techniques will certainly increase in the future. At the same time, it should be considered that these instruments are very expensive and not available in many laboratories.

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Declarations

Conflict of interest The authors declare that they have no conflicts of interest.

Compliance with Ethics requirements This article does not contain any studies with human or animal subjects.

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References

1. Boggia R, Zunin P, Turrini F (2020) Functional foods and food supplements. Appl Sci 10:8538. https://doi.org/10.3390/app10238538
2. Market Research (2020) Global functional foods market growth 2020–2025. https://www.marketresearch.com/LP-Infor
3. Duttaroy AK (2019) In: Bagchi D (ed) Nutraceutical and functional food regulations in the united states and around the World. Academic Press, https://doi.org/10.1016/B978-0-12-816467-9.00019-8.
4. Skendi A, Irakli M, Chatzopoulou P (2017) Analysis of phenolic compounds in Greek plants of Lamiaceae family by HPLC. J Appl Res Med Aromat Plants 6:62–69. https://doi.org/10.1007/jjarmp.2017.02.001
5. Cory H, Passarelli S, Szeto J, Tamez M, Mattei J (2018) The role of polyphenols in human health and food systems: a mini-review. Front Nutr 5:87. https://doi.org/10.3389/fnut.2018.00087
6. Belščak-Cvitanović A, Komes D, Durgo K, Vojvodić A, Bušić A (2015) Nettle (Urtica dioica L.) extracts as functional ingredients for production of chocolates with improved bioactive composition and sensory properties. J Food Sci Technol 52:7723–7734. https://doi.org/10.1007/s13197-015-1916-y
7. Caleja C, Barros L, Barreira JCM, Cricic A, Sokovic M, Calhelha RC, Beatriz M, Oliveira PP, Ferreira ICFR (2018) Suitability of lemon balm (Melissa officinalis L.) extract rich in rosmarinic acid as a potential enhancer of functional properties in cupcakes. Food Chem 250:67–74. https://doi.org/10.1016/j.foodchem.2018.01.034
8. Hasneen DF, Zaki NL, Abbas MS, Ashoush IS, Fayed AE (2020) Comparative evaluation of some herbs and their suitability for skimmed milk yoghurt and cast Kariesh cheese fortification as functional foods. Ann Agric Sci 65:6–12. https://doi.org/10.1016/j.aoas.2020.05.001
9. de Carvalho FAL, Munekata PES, de Oliveira AL, Pateiro M, Domínguez R, Trindade MA, Lorenzo JM (2020) Turmeric (Curcuma longa L.) extract on oxidative stability, physicochemical and sensory properties of fresh lamb sausage with fat replacement by tiger nut (Cyperus esculentus L.) oil. Food Res Int 136:9487. https://doi.org/10.1016/j.foodres.2020.109487
10. Melilli MG, Pagliaro A, Scandurra S, Gentile C, Stefano VD (2020) Omega-3 rich foods: durum wheat spaghetti fortified with Portulaca oleracea. Food Biosci 37:100730. https://doi.org/10.1016/j.fdbiosci.2020.100730
11. Park SJ, Hong SJ, García CV, Lee SB, Shin GH, Kim JT (2019) Stability evaluation of turmeric extract nanoemulsion powder after application in milk as a food model. J Food Eng 259:12–20. https://doi.org/10.1016/j.jfoodeng.2019.04.011
12. Ribeiro A, Caleja C, Barros L, Santos-Buelga C, Barreiro MF, Ferreira ICFR (2016) Rosemary extracts in functional foods: extraction, chemical characterization and incorporation of free and microencapsulated forms in cottage cheese. Food Funct 7:2185–2196. https://doi.org/10.1039/C6FO00270F
13. Ribas-Agustí A, Gratacós-Cubarsí M, Sárraga C, Guàrdia MD, García-Regueiro JA, Castellari M (2014) Stability of phenolic compounds in dry fermented sausages added with cocoa and grape seed extracts. LWT 57:329–336. https://doi.org/10.1016/j.lwt.2013.12.046
14. Fadavi A, Beglaryan R (2015) Optimization of UF-Feta cheese preparation, enriched by peppermint extract. J Food Sci Technol 52:952–959. https://doi.org/10.1007/s13197-013-1051-6
15. Mazur L, Gubskyi S, Dorohovych A, Fayed AE (2020) Comparative evaluation of some herbs and their antioxidant properties of candy caramel with plant extracts. Ukr Food J 7:7–21. https://doi.org/10.24263/2304-974X-2018-7-1-3
16. Pasqualone A, Bianco AM, Paradiso VM, Gambacorta G, Caleja C, Barros L, Santos-Buelga C, Barreiro MF, Ferreira ICFR (2019) Preparation, enriched by peppermint extract. J Food Sci Technol 56:385–393. https://doi.org/10.1016/j.foodres.2014.07.014
17. El-Sayed MI, Ibrahim AA, Awad S (2019) Impact of Purslane (Portulaca oleracea L.) extract as antioxidant and antimicrobial agent on overall quality and shelf life of Greek-style yoghurt.
Egypt J Food Sci 47(51):64. https://doi.org/10.21608/ejfs.2019.12089.1005

18. Komes D, Bušić A, Belščak-Cvitanović A, Brnčić M, Bosiljkov T, Vojvodić A, Dujmić F (2017) Novel approach to the development of functional goat’s milk-based beverage using medicinal plant extracts in combination with high intensity ultrasound treatment. Food Technol Biotechnol 55:484–495. https://doi.org/10.17113/fbb.55.04.17.5123

19. Muhammad DRA, Tuenter E, Patria GD, Foubert K, Pieters L, Dewettinck K (2021) Phytochemical composition and antioxidant activity of Cinnamomum burmannii Blume extracts and their potential application in white chocolate. Food Chem 340:127983. https://doi.org/10.1016/j.foodchem.2020.127983

20. Wihasah RRS, Arief II, Batubara I (2018) Anti-diabetic potency and characteristics of probiotic goat-milk yogurt supplemented with Roselle extract during cold storage. Trop J Anim Sci 41:191–199. https://doi.org/10.5398/tjas.2018.41.3.191

21. Hong H, Lim JM, Kothari D, Kwon SH, Kwon HC, Han SG, Kim SK (2021) Antioxidant properties and diet-related α-glucosidase and lipase inhibitory activities of yogurt supplemented with Safflower (Carthamus tinctorius L.) petal extract. Food Sci Anim Resour 41:122–134. https://doi.org/10.5851/kfsa.2020.e88

22. Ahmed IAM, Alqah HAS, Saleh A, Al-Juhaimi FY, Babiker EE, Ghafoor K, Hassan AB, Osman MA, Fickak A (2021) Physicochemical quality attributes and antioxidant properties of set-type yogurt fortified with argel (Solenostemma argel Hayne) leaf extract. LWT 137:110389. https://doi.org/10.1016/j.lwt.2020.110389

23. Lee NK, Jeewanthisi RKC, Park EH, Paik HD (2016) Short communication: physicochemical and antioxidant properties of rye-type cheese fortified with Inula britannica extract. J Dairy Sci 99:83–88. https://doi.org/10.3168/jds.2015-9935

24. Pasukamonset P, Pulamee T, Sanguansuk N, Chumyen C, Wongvasu P, Adisakwattana S, Ngamukote S (2018) Physicochemical, antioxidant and sensory characteristics of sponge cakes fortified with Clitoria ternatea extract. J Food Sci Technol 55:2881–2889. https://doi.org/10.1007/s13217-018-3204-0

25. Bajerska J, Mildner-Szkudlarz S, Jeszka J, Szwengiel A (2010) Characterization and bio-accessibility evaluation of olive leaf extract-enriched “taralli.” Foods 9:1268. https://doi.org/10.3390/foods901268

26. Cedola A, Palermo C, Centonze D, Nobile MAD, Conte A (2020) Extraction of Salicornia europaea in fresh pasta to enhance phenolic compounds and antioxidant activity. Int J Food Technol 54:3051–3057. https://doi.org/10.1111/ijfs.14218

27. Padalino L, Costa C, Nobile MAD, Conte A (2019) Extract of Salicornia europaea in fresh pasta to enhance phenolic compounds and antioxidant activity. Int J Food Technol 54:3051–3057. https://doi.org/10.1111/ijfs.14218

28. Shori AB, Kee LA, Baba AS (2021) Total phenols, antioxidant activity and sensory evaluation of bread fortified with spear-mint. Arab J Sci Eng 46:5257–5264. https://doi.org/10.1007/s13369-020-05012-5

29. Zhao Y, Kong H, Zhang X, Hu X, Wang M (2019) The effect of Perilla (Perilla frutescens) leaf extracts on the quality of surimi fish balls. Food Sci Nutr 7:1268. https://doi.org/10.1007/s00217-019-00912-8

30. Das AK, Rajkumar V, Verma AK, Swarup D (2012) Moringa oleifera leaves extract: a natural antioxidant for retarding lipid peroxidation in cooked goat meat patties. Int J Food Sci Technol 47:585–591. https://doi.org/10.1111/j.1365-2621.2011.02881.x

31. Jahangir MA, Shehzad A, Butt MS, Bashir S (2018) Influence of supercritical fluid extract of Cinnamomum zeylanicum Bark on physical, bioactive and sensory properties of innovative cinnamon Aliment 17:235–245. https://doi.org/10.17306/J.AFS.0571

32. Abdel-Moemin AR (2016) Effect of Roselle calyces extract on the chemical and sensory properties of functional cupcakes. Food Sci Hum Wellness 5:23–237. https://doi.org/10.1016/j.fshw.2016.07.003

33. Tagliazucchi D, Verzelloni E, Bertolini D, Conte A (2010) In vitro bio-accessibility and antioxidant activity of grape polyphenols. Food Chem 120:599–606. https://doi.org/10.1016/j.foodchem.2009.10.030

34. Sun L, Miao M (2020) Dietary polyphenols modulate starch digestion and glycemic level: a review. Crit Rev Food Sci Nutr 60:541–555. https://doi.org/10.1080/10408398.2018.1544883

35. Ayua EO, Nkhata SG, Namaumbo SJ, Kamau EH, Ngoma TN, Adoul KO (2021) Polyphenolic inhibition of enterocytic starch digestion enzymes and glucose transporters for managing type 2 diabetes may be reduced in food systems. Helioy 7:e06245. https://doi.org/10.1016/j.helyon.2021.e06245

36. Kan L, Oliviero T, Verkerk R, Fogliano V, Capuano E (2020) Interaction of bread and berry polyphenols affects starch digestibility and polyphenols bio-accessibility. J Funct Foods 68:103924. https://doi.org/10.1016/j.jff.2020.103924

37. Coe S, Ryan L (2016) White bread enriched with polyphenol extracts shows no effect on glycemic response or satiety, yet may increase postprandial insulin economy in healthy participants. Nutr Res 36:193–200. https://doi.org/10.1016/j.nutres.2015.10.007

38. Čepo DV, Rudić K, Turićić P, Anić D, Komar B, Šalov M (2020) Food (matrix) effects on bioaccessibility and intestinal permeability of major olive antioxidants. Foods 9:1831. https://doi.org/10.3390/foods9121831

39. Ortega N, Reguant J, Romero MP, Macià A, Motivia MJ (2009) Effect of fat content on the digestibility and bioaccessibility of cocoa polyphenol by an in vitro digestion model. J Agric Food Chem 57:5743–5749. https://doi.org/10.1021/jf900591q

40. Kim DH, Cho WY, Yeon SJ, Choi SH, Lee CH (2019) Effects of Lotus (Nelumbo nucifera) leaf on quality and antioxidant activity of yogurt during refrigerated storage. Food Sci Anim Resour 39:792–803. https://doi.org/10.5851/kfsa.2019.e69

41. Helal A, Tagliazucchi D (2018) Impact of in-vitro gastro-pancreatic digestion on polyphenols and cinnamaldehyde bioaccessibility and antioxidant activity in stirred cinnamon-fortified yogurt. J Agric Food Chem 89:164–170. https://doi.org/10.1021/acs.jafc.7b04074

42. Lamothe S, Azimi N, Bazinet L, Couillard C, Britten M (2014) Interaction of green tea polyphenols with dairy matrices in a simulated gastrointestinal environment. Food Funct 5:2621–2631. https://doi.org/10.1039/c4fo00203b

43. Lončarević I, Pajić B, Petrović J, Zarić Đ, Šaponjac VT, Fišteš A, Jovanović P (2019) The physical properties, polyphenol content and sensory characteristics of white chocolate enriched with black tea extract. Food in Health Dis Sci-Profess J Nut Diet 8:83–88

44. Muhammad DRA, Saputro AD, Rottiers H, de Walle DV, Dewettinck K (2018) Physicochemical properties and antioxidant activities of chocolates enriched with engineered cinnamon nanoparticles. Eur Food Res Technol 244:1185–1202. https://doi.org/10.1007/s00217-018-3035-2

45. Gómez-Mascareña LG, Hernández-Rojas M, Tarancón P, Tenon M, Feuillère N, Ruiz JFV, Fiszman S (2017) Impact of microcapsulation within electrosprayed proteins on the formulation of cinnamaldehyde-enriched chocolates. Czech J Food Sci 36:28–36. https://doi.org/10.17221/237/2016-CJFS

46. El-Said MM, El-Messery T, El-Din HM (2018) The encapsulation of powdered doum extract in liposomes and its application in yogurt. Acta Sci Pol Technol Aliment 17:235–245. https://doi.org/10.17306/J.AFS.0571
47. Curren MSS, King JW (2002) In: Pawliszyn J (ed) Comprehensive analytical chemistry. Elsevier Inc, https://doi.org/10.1016/S0166-526X(02)80062-9

48. Socas-Rodríguez B, Asensio-Ramos M, Hernández-Borges J, Herrera-Herrera AV, Rodríguez-Delgado MA (2013) Chromatographic analysis of natural and synthetic estrogens in milk and dairy products. TrAC-Trend Anal Chem 44:58–77. https://doi.org/10.1016/j.trac.2012.10.013

49. Rostagno MA, D’Arrigo M, Matinez JA (2010) Combinatory and hyphenated sample preparation for the determination of bioactive compounds in foods. TrAC-Trend Anal Chem 29:553–561. https://doi.org/10.1016/j.trac.2010.02.015

50. Belščak-Cvitanović A, Komes D, Benković M, Karlović S, Hečimović I, Ježek D, Bauman I (2012) Innovative formulations of chocolates enriched with plant polyphenols from Rubus idaeus L. leaves and characterization of their physical, bioactive and sensory properties. Food Res Int 48:820–830. https://doi.org/10.1016/j.foodres.2012.06.023

51. Đurović S, Vujanović M, Radijović M, Filipović J, Filipović G, Gašić U, Tešić Ž, Mašković P, Zeković Z (2020) The functional food production: application of stinging nettle leaves and its extracts in the baking of a bread. Food Chem 312:126091. https://doi.org/10.1016/j.foodchem.2019.126091

52. Dordoni R, Garrido GD, Marinoni L, Torri L, Piochi M, Spigno L. leaves and characterization of their physical, bioactive and sensory properties. Food Res Int 48:820–830. https://doi.org/10.1016/j.foodres.2012.06.023

53. Rastogi S, Katara A, Pandey MM, Arora S, Singh RRB, Rawat AKS (2013) Physical stability and HPLC analysis of Indian Kudzu (Pueraria tuberosa Linn.) fortified milk. Evid-Based Complement Altern Med. 15:10. https://doi.org/10.1155/2013/368248

54. Gramza-Michałowska A, Kubus-Cisyńska J, Kmietick D, Korczak J, Helak B, Dziedzic K, Górecka D (2016) Antioxidative potential, nutritional value and sensory profiles of confectionery fortified with green and yellow tea leaves (Camellia sinensis). Food Chem 211:448–454. https://doi.org/10.1016/j.foodchem.2016.05.048

55. Didar Z (2020) Characterization of white chocolate enriched with free or encapsulated pomegranate extract. J Nutr Fast Health 8:302–309. https://doi.org/10.22038/jnfh.2020.50603.1281

56. Singh B, Kumar A, Malik AK (2014) Recent Advances in sample preparation methods for analysis of endocrine disruptors from various matrices. Crit Rev Anal Chem. 44:255–269. https://doi.org/10.1080/10408347.2013.859981

57. Jang S, Xu Z (2009) Lipophilic and hydrophilic antioxidants and their antioxidant activities in purple rice bran. J Agric Food Chem 57:858–862. https://doi.org/10.1021/jf803113c

58. Martini S, Conte A, Tagliasacchi D (2018) Comprehensive evaluation of phenolic profile in dark chocolate and dark chocolate enriched with Sakura green tea leaves or turmeric powder. Food Res Int 112:1–16. https://doi.org/10.1016/j.foodres.2018.06.020

59. Zhang Y, Bao B, Lu B, Ren Y, Tie X, Zhang Y (2005) Determination of flavor C-glucosides in antioxidant of bamboo leaves (AOB) fortified foods by reversed-phase high-performance liquid chromatography with ultraviolet diode array detection. J Chrom A 1065:177–185. https://doi.org/10.1016/j.chroma.2004.12.086

60. Lea A (2008) In: Ottaway PB (ed) Food fortification and supplementation. CRC Press, https://doi.org/10.1533/9781845694265.2.175

61. Qiu X, Jacobsen C, Sørensen ADM (2018) The effect of rosemary (Rosmarinus officinalis L.) extract on the oxidative stability of lipids in cow and soy milk enriched with fish oil. Food Chem 263:119–126. https://doi.org/10.1016/j.foodchem.2018.04.106

62. Melilli MG, Stefano VD, Sciacca F, Pagliaro A, Bognanni R, Scandurra S, Vrizi N, Gentile C, Palumbo M (2020) Improvement of fatty acid profile in durum wheat breads supplemented with Portulaca oleracea L. quality traits of purslane-fortified bread. Foods 9:764. https://doi.org/10.3390/foods9060764

63. Sik B, Lakatos EH, Kacsándi V, Székelyhidi R, Zs A (2021) Exploring the rosmarinic acid profile of dark chocolate fortified with freeze-dried lemon balm extract using conventional and non-conventional extraction techniques. LWT 147:111520. https://doi.org/10.1016/j.lwt.2021.111520

64. Belwal T, Ezzat SM, Rastrelli L, Bhatt ID, Daglia M, Baldi A, Devonka HP, Orhan IE, Patra JK, Das G, Anandhamarkrishnan C, Gomez-Gomez L, Nabvi SF, Nabvi SM, Atanasov AG (2018) A critical analysis of extraction techniques used for botanicals: trends, priorities, industrial uses and optimization strategies. TrAC-Trend Anal Chem 100:82–102. https://doi.org/10.1016/j.trac.2017.12.018

65. Bagade SB, Patil M (2019) Recent advances in microwave assisted extraction of bioactive compounds from complex herbal samples: a review. Crit Rev Anal Chem 15:1–12. https://doi.org/10.1080/10408347.2019.1686966

66. Dzah CS, Duan Y, Zhang H, Wen C, Zhang J, Chen G, Ma H (2020) The effect of ultrasound assisted extraction on yield, antioxidant, anticancer and antimicrobial activity of polyphenol extracts: a review. Food Biosci 35:100546. https://doi.org/10.1016/j.foodiobio.2020.100547

67. Lefebvre T, Destandau E, Lesellier E (2020) Selective extraction of bioactive compounds from plants using recent extraction techniques: a review. J Chrom A 1635:461770. https://doi.org/10.1016/j.chroma.2020.461770

68. Hayat HI, Khokha M, El-hocine S, Halim B, Mansouri H, Nawel A, Khodir M, Lila BM (2021) Effect of rosemary (Rosmarinus officinalis L.) supplementation on fresh cheese: physicochemical properties, antioxidant potential and sensory attributes. J Food Process Preserv 45:e15057. https://doi.org/10.1111/jfpp.15057

69. Servili M, Rizzello CG, Taticchi A, Esposto S, Urbani S, Mazzacane F, Di Maio I, Selvaggini R (2011) Functional milk beverage fortified with phenolic compounds extracted from olive vegetation water, and fermented with functional lactic acid bacteria. Int J Food Microbiol 147:45–52. https://doi.org/10.1016/j.ijfoodmicro.2011.03.006

70. Romeo R, Bruno AD, Imeneo V, Piscopo A, Poiana M (2020) Impact of stability of enriched oil with phenolic extract from olive mill wastewaters. Foods 9:856. https://doi.org/10.3390/foods9070856

71. Banerjea V, Stokanović MC, Flanjak I, Doko K, Jozinović A, Babić J, Šubarić D, Miličević B, Čindrić I, Ačkar D (2020) Coca shell as a step forward to functional chocolates—bioactive components in chocolates with different composition. Molecules 25:5470. https://doi.org/10.3390/molecules25225470

72. Cho WY, Kim DH, Lee HJ, Yeon SJ, Lee CH (2019) Quality characteristic and antioxidant activity of yogurt containing olive leaf hot water extract. CyTA J Food 18:43–51. https://doi.org/10.1080/19476337.2019.1640797

73. Baiano A, Viggian I, Terracene C, Romaniello R, Del Nobile MA (2015) Physical and sensory properties of bread enriched with phenolic aqueous extracts from vegetable wastes. Czech J Food Sci 33:247–253. https://doi.org/10.17221/528/2014-CJFS

74. Setyaningsih W, Saputro IE, Palma M, Barroso CG (2016) Stability of 40 phenolic compounds during ultrasound-assisted extractions (UAE). AIP Conf Proc 1755:080009. https://doi.org/10.1063/1.4958517

75. Mondal A, Datta AK (2008) Bread baking—a review. J Food Eng 86:465–474. https://doi.org/10.1016/j.jfoodeng.2007.11.014

76. Feng C, Wang B, Zhao A, Wei L, Shao Y, Wang Y, Cao B, Zhang F (2019) Quality characteristics and antioxidant activities of goat milk yogurt with added jujube pulp. Food Chem 277:238–245. https://doi.org/10.1016/j.foodchem.2018.10.104
77. Raikos V, Ni H, Hayes H, Ranawana V (2019) Antioxidant properties of a yogurt beverage enriched with salal (Gaultheria shallon) berries and blackcurrant (Ribes nigrum) pomace during cold storage. Beverages 5:2. https://doi.org/10.3390/beverages5010002

78. Bhat NA, Hamdani AM, Masoodi FA (2018) Development of functional cookies using saffron extract. J Food Sci Technol 55:4918–4927. https://doi.org/10.1007/s13197-018-3426-1

79. Oh NS, Lee JY, Joung JY, Kim KS, Shin YK, Lee KW, Kim SH, Oh S, Kim Y (2016) Microbiological characterization and functionality of set-type yogurt with potential prebiotic substrates Cudrania tricuspidata and Morus alba L. leaf extracts. J Dairy Sci 9:6014–6025. https://doi.org/10.3168/jds.2015-10814

80. Chan CL, Gan RY, Shah NP, Corke H (2018) Enhancing antioxidant capacity of Lactobacillus acidophilus-fermented milk fortified with pomegranate peel extracts. Food Biosci 6:185–192. https://doi.org/10.1016/j.fbio.2018.10.016

81. Righetti L, Paglia G, Galaverna G, Dall’Asta C (2011) Recent advances and future challenges in modified mycotoxin analysis: why HRMS has become key instrument in food contaminant research. Toxins 8:361. https://doi.org/10.3390/toxins8120361

82. Kaufmann A (2012) The current role of high-resolution mass spectrometry in food analysis. Anal Bioanal Chem 403:1233–1249. https://doi.org/10.1007/s00216-011-5629-4

83. Lucci P, Saurina J, Núñez O (2017) Trends in LC-MS and LC-HRMS analysis and characterization of polyphenols in food. TrAC Trend Anal Chem 88:1–24. https://doi.org/10.1016/j.trac.2016.12.006

84. Allen DR, McWhinney BC (2019) Quadrupole time-of-flight mass spectrometry: a paradigm shift in toxicology screening applications. Clin Biochem Rev 40:135–146. https://doi.org/10.33176/AACB-19-00023

85. Szołtysik M, Kucharska AZ, Sokół-Łętowska A, Dąbrowska A, Bobak Ł, Chrzanowska J (2020) The effect of Rosa spinosissima fruits extract on lactic acid bacteria growth and other yoghurt parameters. Foods 9:1167. https://doi.org/10.3390/foods9091167

86. Mikulec A, Kowalski S, Makarewicz M, Skoczylas L (2020) Cistus extract as a valuable component for enriching wheat bread. LWT 118:108713. https://doi.org/10.1016/j.lwt.2019.108713

87. Cedeño-Pinos C, Martínez-Tomé M, Murcia MA, Jordán MJ, Bañón S (2020) Assessment of rosemary (Rosmarinus officinalis L.) extract as antioxidant in jelly candies made with fructan fibres and stevia. Antioxidants 9:1289. https://doi.org/10.3390/antiox9121289

88. Yadav K, Bajaj RK, Mandal S, Saha P, Mann B (2017) Evaluation of total phenol content and antioxidant properties of encapsulated grape seed extract in yoghurt. Int J Dairy Technol 71:96–104. https://doi.org/10.1111/1471-0307.12464

89. Wang R, Zhou W (2004) Stability of tea catechins in the breadmaking process. J Agric Food Chem 52:8224–8229. https://doi.org/10.1021/jf040865x

90. Ignat I, Volî I, Popa VI (2013) In: Ramawat KG, Méronn IM (eds) Natural products. Springer, https://doi.org/10.1007/978-3-642-22144-6_56

91. Karamaslan M, Ozden M, Vardin H, Turkoglu H (2011) Phenolic fortification of yogurt using grape and callus extracts. LWT 44:1065–1072. https://doi.org/10.1016/j.lwt.2010.12.009

92. Salta FN, Mylona A, Chiou A, Boskou G, Andrikopoulos NK (2007) Oxidative stability of edible vegetable oils enriched in polyphenols with olive leaf extract. Food Sci Technol Int 13:413–421. https://doi.org/10.1177/1082013208089563

93. Aguiar J, Gonçalves J, Alves VL, Câmara JS (2020) Chemical fingerprint of free polyphenols and antioxidant activity in dietary fruits and vegetables using a non-target approaches based on QuEChERS ultrasound-assisted extraction combined with UHPLC-PDA. Antioxidants 9:305. https://doi.org/10.3390/antiox9040305

94. Casado N, Perestrello R, Silva CL, Sierra I, Câmara JS (2018) An improved and miniaturized analytical strategy based on μ-QuEChERS for isolation of polyphenols. A powerful approach for quality control of baby foods. Microchem J 139:110–118. https://doi.org/10.1016/j.microm.2018.02.026

95. Galarce-Bustos O, Novoa L, Pavon-Perez J, Henriquez-Aedo K, Aranda M (2019) Chemometric optimization of QuEChERS extraction method for polyphenol determination in beers by liquid chromatography with ultraviolet detection. Food Anal Methods 12:448–457. https://doi.org/10.1007/s12161-018-1376-x

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