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REVIEW ARTICLE

Recent advances and comparisons of conventional and alternative extraction techniques of phenolic compounds

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Abstract Phenolic compounds are a group of secondary metabolites produced by plants under stressful conditions. Phenolic compounds play an important role in the prevention and treatment of certain illnesses and are exploited by the food and pharmaceutical industries. Conventional methods are commonly used as models to compare the efficiencies of alternative extraction methods. Among alternative extraction processes, microwave-assisted extraction (MAE), pressurized liquid extraction (PLE), supercritical fluid extraction (SFE) and ultrasonic-assisted extraction (UAE) are the most studied. These methods produce extracts rich in phenolic compounds using moderate temperatures, short extraction times, and solvents generally recognized as safe. The combination of extraction time and temperature plays a critical role in the stability of the compounds. Solvents of higher polarity enhance the extraction of phenolic compounds. The use of the ethanol–water mixture for MAE, PLE, and UAE is recommended. MAE and UAE involve shorter extraction times than do PLE and SFE. SFE requires a low average temperature (40 °C). MAE produces the highest total phenolic content [227.63 mg GAE/g dry basis (d.b.)], followed by PLE (173.65 mg GAE/g d.b.), UAE (92.99 mg GAE/g d.b.) and SFE (37 mg GAE/g d.b.). Extraction yields and recovery rates of the phenolic compounds can be enhanced by combining and integrating extraction methods.

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Introduction

Phenolic compounds are a ubiquitous group of secondary metabolites in fruits and vegetables. Experts recommended a diet rich in fruits and vegetables in part because they are an important source of phenolic compounds, which play an important role in the prevention and treatment of certain illnesses (Luna-Guevara et al. 2018). For instance, phenolic compounds are widely used as natural antioxidants and antimicrobial agents (Tanase et al. 2019). Phenolic compounds also exhibit antiallergenic, antiatherogenic, and anti-inflammatory activities that can be exploited by food and pharmaceutical industries. As phenolic compounds are found in all plants, almost all research related to the extraction of phenolic compounds focuses on bioprospecting for new plant varieties to serve as sources of these compounds. Phenolic compounds could be valuable components of products that decrease the risk of cardiovascular and neurological diseases as well as cancer. Meanwhile, the antioxidant power of phenolic compounds suggests they could be used as natural additives to functional foods. For example, phenolic compounds from olive mill wastewater were able to prolong the shelf life of bakery products due to its antimicrobial properties (Galanakis et al. 2018). In the same way, Basanta et al. (2018) developed a colored film containing phenolics compounds extracted from cherry that constituted a food preserving antioxidant barrier.

However, the extraction of these compounds is challenging because they can be unstable and biological

activity can be affected by both extraction process parameters and external factors such as the presence of oxygen and light. Alternative and more efficient extraction processes are required to overcome these drawbacks and produce higher extraction yields while maintaining compound integrity. Among the alternative extraction processes, microwave-assisted extraction (MAE), pressurized liquid extraction (PLE), supercritical fluid extraction (SFE), and ultrasonic-assisted extraction (UAE) are the most studied. These processes are recognized as environmentally friendly and are associated with short extraction times and low solvent consumption rates. MAE is a sustainable technology for the extraction of phenolic compounds in which microwaves break up the cellular matrix, releasing intracellular compounds. This technique uses polar solvents that easily absorb microwave energy and enhance the extraction process (De Castro and Castillo-Peinado 2016). PLE uses liquid solvents at temperatures above their atmospheric boiling points, but below their critical points, enhancing solubility and mass transfer properties. SFE also takes advantage of high pressures, using a gas above its critical point to extract bioactive compounds. In this technique, the most commonly used solvent is carbon dioxide because, besides being nontoxic and nonflammable, it is readily available at reasonable low cost. In UAE, an acoustic phenomenon known as cavitation enhances mass transfer rates and solvent penetration, producing higher extraction yields and short extraction times. This review compares conventional and alternative methods of extracting phenolic compounds, and compares alternative extraction methods against each other. The first section presents the characteristics of phenolic compounds. Next, the extraction of phenolic compounds by conventional and alternative methods is described, along with the most important extraction parameters and recent studies. Finally, a comparison of the process parameters, yield, and efficiency involved in each extraction technique is presented.

Phenolic compounds

Among the secondary metabolites produced by plants, phenolic compounds are one of the more abundant groups. For example, more than 800 types of phenolic compounds have been identified in plants (Cvejic et al. 2017). Chemically, phenolic compounds have at least one aromatic ring and one or more hydroxyl groups. The ability to chelate metals is primarily responsible for its antioxidant activity. Generally, phenolic compounds are most often classified as either flavonoids and non-flavonoids. Their basic structure is presented in Table 1. Flavonoids, the most abundant plant polyphenols consumed by humans, are polyphenolic compounds with two aromatic rings linked by a three-carbon bridge (Roleira et al. 2015). Flavonoids are assigned into one of six families, according to differences in the aromatic ring: flavones, isoflavones, flavonols, flavanones, flavan-3-ols, and anthocyanidins (Table 1). Non-flavonoids are relatively smaller and simpler than flavonoids or have complex structures with high molecular weights. Among fruits and vegetables, phenolic acids are recognized as the most important family of non-flavonoids. Phenolic acids can be divided into three families: hydroxybenzoic acids, hydroxycinnamic acids, and other hydroxyphenyl acids (De la Rosa et al. 2019).

Although phenolic compounds are not essential for human metabolism, if they are present in a diet, they can improve health and may reduce the risk of serious diseases. In fact, phenolic compounds are widely studied because they are able to reduce the reactive oxygen species that cause oxidative stress (Socrier et al. 2019) and are associated with the prevention and treatment of cardiovascular (Yousefian et al. 2019) and chronic degenerative diseases (Luna-Guevara et al. 2018). Moreover, phenolic compounds are recognized for their antibacterial (Ullah et al. 2019), anti-inflammatory (Liu et al. 2018a, b), anti-diabetic (Chen et al. 2019) and anti-cancer (Martini et al. 2018) properties.

As can be seen in Table 1, phenolic compounds are found in several edible plants. They can contribute to organoleptic properties and they make a paramount contribution to maintaining the oxidative stability of foods (Cvejic et al. 2017). Phenolic compounds can therefore be used as natural preservatives or as part of bioactive packaging films. Thus, extraction is a key factor in the use and incorporation of these compounds in foods and their components.

Conventional extraction methods

Phenolic compounds have been extracted for decades using conventional extraction methods such as Soxhlet, maceration, infusion, and digestion (Wong-Paz et al. 2017). Soxhlet extraction and maceration are the most common techniques. For example, the extraction of phenolic compounds using maceration and Soxhlet from grape skin (Caldas et al. 2018), *Vernonia cinerea* leaves (Alara et al. 2018a, b, c) and feijoa peel (Henrique et al. 2019), produces a total phenolic content (TPC) of between 48.6 and 71 mg of gallic acid equivalent (GAE) per gram.

Soxhlet extraction and maceration generally use high solvent/feed (S/F) ratios (above 20) and long extraction times for exhaustive extraction of all compounds from a matrix. Thus, these methods are commonly used as models to compare the efficiencies of alternative methods.

Table 1 Phenolic compounds families, major compounds and mainly sources. Based on Tsimogiannis and Oreopoulou (2019)

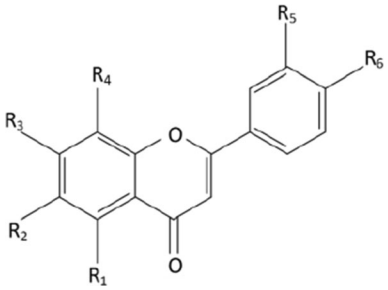
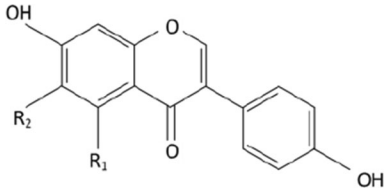
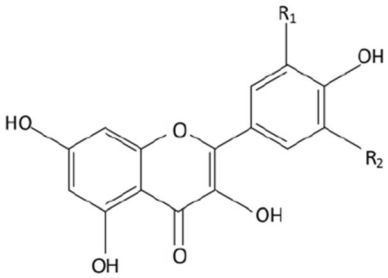
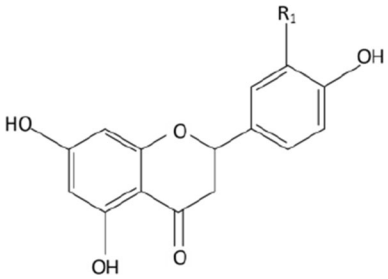
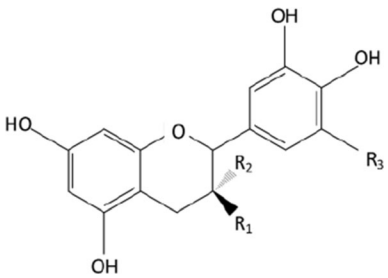
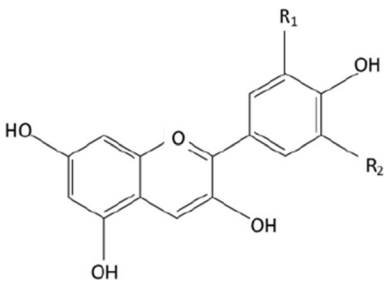
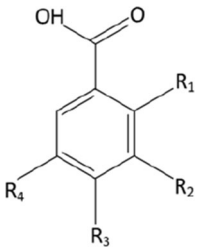
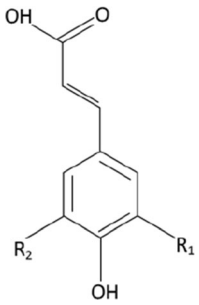
Family	Basic structure	Major compounds	Mainly source
Flavones		Apigenin, luteolin, tangeretin, Nobiletin	Celery, parsley, thyme, cantaloupe, water-melon, citrus peels and juices, sweet and hot peppers, Chinese cabbage and artichokes
Isoflavones		Daidzein, genistein, glycitein	Soy beans, legumes, beans and peanuts
Flavonols		Kaempferol, quercetin, myricetin	Grapes, cherries, artichokes, Chinese cabbage, hot peppers, lettuce, onion, walnut and herbs from the apiaceae family
Flavanones		Naringenin, eriodictyol, hesperitin	Citrus fruits and juices, Mexican oregano and pepper-mint
Flavan-3-ols		Catechin, epicatechin, gallocatechin, epigallocatechin, catechin gallate, epicatechin gallate, gallocatechin gallate, epigallocatechin gallate	Tea, chocolate, red wine, nuts, grape, strawberry, blackberry, peach, nectarine, apple, cereals, peach, nectarine, plum, and apple
Anthocyanidins		Pelargonidin, cyaniding, delphinidin, peonidin, petunidin, malvidin	Grapes, cherries, plum, nectarine, peach, black beans, red lettuce and red onion

Table 1 continued

Family	Basic structure	Major compounds	Mainly source
Hydroxybenzoic acids		Salicylic acid, p-hydroxybenzoic acid, protocatechuic acid, vanillic acid, gallic acid and syringic acid	Berries, nuts, tea, chicory, and some spices
Hydroxycinnamic acids		p-coumaric acid, caffeic acid, and the methylated forms ferulic and sinapic acids	Plum, berries, nectarine, peach, apple, pear, broccoli, tomato, chicory, lettuce, olives, carrot and cereals

Extraction times of up to 360 and 720 min have been used for extraction of phenolic compounds using Soxhlet extraction and maceration, respectively.

In Soxhlet extraction, a dry sample is placed in a thimble. The thimble and the solvent are then placed in a distillation flask, which is heated to evaporate the solvent. The condensate and reflux return to the thimble-holder until they reach an overflow level and can be aspirated by a siphon. The compounds are extracted to the bulk liquid. As the flask is continuously heated, the solvent is continually refluxed and the compounds remain in the flask. The reflux process is repeated several times until extraction is complete (Azmir et al. 2013). One of major advantages of Soxhlet extraction is that, unlike maceration, the matrix can be in contact with fresh solvent repeatedly and, as the sample is packed in a thimble, extract filtration is not necessary. However, as the extraction of phenolic compounds is performed using longer extraction times and higher temperatures, the bioactivity of the extracts is diminished due to the degradation of the compounds.

In maceration, the raw material is extracted in a specific solvent for a period of time. Maceration can be performed with or without agitation. Contrary to Soxhlet extraction, maceration is performed using lower temperatures. For example, the extraction of phenolic compounds from oil mixtures by maceration is performed under optimized conditions at room temperature (Ji et al. 2018). Thus, the main advantages of maceration include that the process is performed using low temperatures and as the equipment required is not complicated, the process is inexpensive. However, maceration has lower yields and it uses longer extraction times.

Alternative extraction methods

Unlike conventional methods, alternative methods can produce extracts rich in phenolic compounds using moderate temperatures with shorter extraction times and solvents generally recognized as safe. Although the nature and properties of the raw materials, including variety, cultivar, and maturity stage strongly influence the extraction of phenolic compounds, all extraction processes share some major parameters. Choice of solvent, temperature, and extraction time can exhibit similar behavior in all extraction processes. The solubility of phenolic compounds is higher in polar solvents such as water and ethanol or their mixtures, the diffusion of compounds and mass transfer rates are enhanced by increased temperature, and longer extraction times allow for a more intimate and effective contact between solvent and matrix.

Microwave-assisted extraction

MAE is an efficient technique due to its ability to heat a matrix internally and externally without a thermal gradient

(Calle and Costas-Rodriguez 2017). Molecules with a permanent dipole moment such as phenolic compounds and ionic solutions strongly absorb microwave energy. Moreover, microwaves cause internal superheating of water molecules of a sample, promoting cellular disruption and enhancing the recovery of target compounds from the matrix (Yahya et al. 2018).

In microwave systems, the powdered sample is mixed with a measured quantity of extraction solvent before the mixture is loaded into the equipment. The energy can be applied randomly or focused. When microwave radiation is randomly dispersed, the system is considered multi-mode, whereas when microwave radiation is focused on a restricted zone, it is considered single mode. Multi-mode is typically a closed system associated with high pressures while single mode is an open system employed under atmospheric operating pressure. Both systems share four basic components: a magnetron, a waveguide, an applicator, and the circulator (De Castro and Castillo-Peinado 2016).

Process parameters such as solvent choice, temperature, microwave power, and extraction time are crucial influences in MAE. Generally, extraction yields in MAE increase as microwave power increases due to localized heating, which contributes to the rupture of the matrix. However, there is a limit above which microwave power can cause a decrease in extraction yield. This behavior was observed by Alara et al. (2017) in the extraction of phenolic compounds from *Vernonia amygdalina* leaves. In that work, extraction increased when microwave power increased from 400 to 500 W. However, when power exceeded 500 W, a significant decline in the total flavonoid content and antioxidant activity was observed. Generally, overexposure to microwave radiation leads to overheating and the degradation of phenolic compounds, reducing bioactivity and decreasing extraction yields.

As show in Table 2, solvents such as water, ethanol, methanol, acetone, and their mixtures can effectively extract phenolic compounds using MAE. Among them, water–ethanol mixtures are the most commonly used in the recovery of phenolic compounds. According to Morerira et al. (2017), the efficiency and selectivity of MAE depends on the dielectric constant of the solvent mixture. The polarity of organic solvents is increased by the addition of water, the temperature inside the sample is increased due to improved absorption of microwave energy, and the extraction of phenolics increases. When a water–ethanol mixture was used to extract phenolic compounds from *Hibiscus sabdariffa* (Pimentel-Moral et al. 2018), a higher amount of phenolic compounds was obtained with intermediate values of percentage of ethanol (45:55 ethanol:water). In similar research conducted by Marić et al. (2018), MAE was used to extract phenolic compounds from two *Lamiaceae* species. The experiment was performed to optimize the effects of concentration of ethanol, temperature, and extraction time on TPC. Solvent concentration and temperature had the greatest influence on phenolic extraction, while ethanol concentration had a negative effect on TPC.

Temperature is another crucial parameter in MAE. As can be seen in Table 2, temperatures between 30 and 180 °C have been used to extract phenolic compounds by MAE. Generally, yield and TPC increase when the extraction temperature is raised. This behavior was observed by Alara et al. (2017) when extracting phenolic compounds from *V. amygdalina* leaves. However, when temperatures above 100 °C were tested, a slight decrease in TPC was observed. Contrary to this study, optimal extraction temperatures of up to 150 and 164 °C have been used in MAE of *Thymus fontanesii* aerial parts (Nabet et al. 2019) and *H. sabdariffa* calyces (Pimentel-Moral et al. 2018), respectively. The stability of the phenolic compounds is related to the interaction among the raw material characteristics and the process parameters (e.g., extraction time, solvent, microwave power, etc.). Thus, the selection of the extraction temperature should be made according to the interaction of these factors.

Pressurized liquid extraction

In this extraction process, temperatures above the solvent boiling point but below the critical point can increase the kinetics of extraction while applying high pressures to maintain the solvents in their liquid state (Panja 2017). In a basic PLE setup, first, the sample is packed into an extractor. The solvent is then pumped into the extractor using a liquid pump and passes through a heating system to reach the desired temperature. To maintain temperatures, the extractor should have a heating jacket. PLE can be carried out in either a static or a dynamic mode. Static extraction is a batch process in which the extractor is pressurized while the outlet valve is kept closed. The valve is then opened and the extract collected. Contrary to the static mode, in the dynamic mode, the outlet valve is kept open and the solvent is pumped continuously through the extractor (Plaza and Turner 2017).

A literature survey on the use extraction of phenolic compounds using PLE (Table 3) revealed that these compounds are extracted at temperatures between 40 and 275 °C and pressures between 10 and 200 bars. In PLE, when the temperature is increased, the solubility of the compounds is enhanced. For example, TPC increases with temperature in PLE due to an increase in diffusion of phenolic compounds into the solvent, favoring their transport (Bursa et al. 2018; Otero et al. 2019; Vitor et al. 2019).

Table 2 Summary of the recently works published on the extraction of phenolic compounds from natural matrices by MAE

Extraction conditions								
Matrix	Total phenolic content	Solvent	Temperature (°C)	Microwave power (W)	Irradiation time (min)	S/F	Optimized parameters	References
Pomegranate peels	199.4 mg GAE/g d.b	Ethanol:water 50:50–70:30 Methanol:water 50:50–70:30	–	100–600	0.5–15	10–60	600 W, ethanol:water 50:50, S/F 60	Kaderides et al. (2019)
Lime peel waste	53 mg GAE/g d.b	Ethanol:water 50:50–100:0	60	140–720	0.15–0.75	20	0.75 min, 140 W, ethanol:water 55:45, 8 cycles	Rodsamran and Sothornvit (2019)
Hibiscus sabdariffa calyces	70.53 mg GAE/g d.b	Water	40–80	300–700	1–10	10–18	3 min, 50 W, 60 °C, S/F 14	Alara and Abdurahman (2019)
Vernonia cinerea leaves	85.64 mg GAE/g d.b	Ethanol:water 20:80–60:40	40	400–600	1–5	10–18	2 min, 444 W, Ethanol:water 47:53, S/F 1:14	Alara et al. (2018a, b, c)
Apple tree wood residues	47.7 mg GAE/g d.b	Ethanol:water 20:80–80:20	60–120	1500	3–37	22–250	20 min, ethanol:water 60:40, S/F 200, 100 °C	Morerira et al. (2017)
Scirpus holoschoenus rhizomes	30.70 mg GAE/g d.b	Acetone:water 0:100–90:10	–	300–900	0.5–2	20	1.15 min, 600 W, acetone:water 56:44	Oussaid et al. (2018)
Origanum glandulosum and *Thymus fontanesii* aerial parts	*O. glandulosum*: 311.36 *T. fontanesii: 227.63* mg GAE/g extract	Ethanol:water 0:100–100:0	30–150	850	1–10	20	*O. glandulosum*: 2 min, ethanol:water 0:100, 42 °C *T. fontanesii*: 9.5 min, ethanol:water 50:50, 150 °C	Nabet et al. (2019)
Vernonia amygdalina leaves	102.24 mg GAE/g d.w	Water	70–110	400–700	2–20	8–16	8 min, 416 W, 100 °C, S/F 8	Alara et al. (2018a, b, c)
H. sabdariffa calyces	14.4251 mg QE/ g	Ethanol:water 15:85–75:25	50–150	1500	5–20	10	12.5 min, 164 °C, Ethanol:water 45:55	Pimentel-moral et al. (2018)
Ascophyllum nodosum, Laminaria japonica, Lessonia trabeculate and Lessonia nigrecens	139.80 GAE mg/100 g d.b	Methanol:water 70:30	110	2.45 GHz	10	10	Best antioxidant activity: *Ascophyllum nodosum*	Yuan et al. (2018a, b)

GAE, gallic acid equivalents; QE, quercetin equivalents; CQAE, caffeoylquinic acid equivalent

The use of elevated pressure, however, maintains the solvent below its boiling point. The solvent penetrates into a matrix more efficiently under elevated pressures and temperature reduces its viscosity and surface tension, facilitating the extraction of compounds located in the internal pores. However, when high pressures are applied,

Table 3 Summary of the recently works published on the extraction of phenolic compounds from natural matrices by PLE

Extraction conditions								
Matrix	Total phenolic content	Solvent	Temperature (°C)	Pressure (bar)	Extraction time (min)	S/F	Optimized parameters	References
Pacific oyster	0.72 g/ 100 g d.b	Water	125–275	20–140	5	30	225 °C, 120 bar	Lee et al. (2018)
Hancornia speciosa	347 mg/g extract	Hexane Ethyl acetate Ethanol:water	25–60	100	180	18	60 °C, ethanol	Barbosa et al. (2019)
Pistachio hulls	39.5 g/kg d.b	Water	110–190	69	480	60	110–150 °C	Erşan et al. 2018)
Orange peel	15.9 mg GAE/g d.b	Ethanol:water 50:50–99.5:0.5	45–65	100	40	47	65 °C, ethanol:water 75:25	Barrales et al. (2018)
Goldenberry pulp	–	Ethanol:water 70:30	25	100–200	10–60	20–60	10 min	Osmar et al. (2018)
Cocoa bean shell	5.1 mg/g	Ethanol	60–90	100	5–50	3	90 °C, 50 min	Okiyama et al. (2018)
Grape (*Vitis vinifera* L. CV. Syrah) marc	65.68 mg GAE/g d.b	Ethanol:water 50:50–100:0 Ethanol:water (pH 2) 50:50 Acidified water (pH 2)	40–100	100	240	217–253	40 °C, Ethanol:water pH 2.0 0% 50:50	Vitor et al. (2019)
Stevia rebaudiana Bertoni leaves	7.41 mg GAE/g	Water	100–160	100	5–10	–	160 °C, 10 min	Bursa et al. (2018)
Barley and canola straws	45.4 mg GAE/g (barley) 52.9 mg GAE/g (canola)	Ethanol:water 20:80–100:0	140–220	50–200	40	–	180 °C, 50 bar, Ethanol:water 20:80	Huerta and Saldaña (2018)
Laminaria ochroleuca	173.65 mg GAE/g extract	Hexane, ethanol, ethyl acetate and ethanol 50%	80–160	100	–	20	160 °C, Ethanol:water 50:50	Otero et al. (2019)
Feijoa peel	132 mg GAE/ g	Ethanol:water 100:0–100:0	40–80	100	–	–	Ethanol:water 50:50, 80 °C	Henrique et al. (2019)

GAE, gallic acid equivalents; QE, quercetin equivalents; CQAE, caffeoylquinic acid equivalent

raw materials can be compacted, which avoids the correct contact between matrix and solvent and diminishes recovery of phenolics compounds.

Supercritical fluid extraction

SFE uses fluids above critical pressures and temperatures. In the region above the critical point, the supercritical fluid has a high solvation power due to their relatively high density (Rosa et al. 2009). Through small variations in system pressure or temperature it is possible to change the density and selectivity of the solvent as well as the solubility of the compounds. In this extraction process, solvent separation is relatively straightforward. A common separation process involves decreasing the pressure of the mixture that leaves the extraction column. The mixture expands in a collection vessel and the solvent is emitted to the atmosphere or re-circulated to the system while the extract in recovered in the vessel.

The extraction process involves packing the sample into an extractor. After which CO_2 is cooled using a cooling bath and pressurized using a liquid pump. CO_2 reaches its

supercritical point after being heated at the desired temperature in a heating bath and directed into the extractor vessel. If a co-solvent is required, it can be injected into the extractor using a liquid pump to achieve the required proportion or pumped simultaneously with the CO_2 into the extractor before the static time. As in PLE, SFE is initially performed using a static time to equilibrate the system and in a dynamic extraction time. Supercritical extraction is then performed and the CO_2 + co-solvent + extract mixture is recovered through decreasing pressure and temperature (Hatami et al. 2018). After the extraction, if a co-solvent was used, it can be evaporated in a rotary evaporator.

Carbon dioxide is the most used supercritical fluid for extracting phenolic compounds (Table 4). Supercritical carbon dioxide ($scCO_2$) has a low critical temperature (31.1 °C) and mild critical pressure (73.8 bars). In addition, $scCO_2$ is nontoxic, noncorrosive, inexpensive, nonflammable, environmentally friendly, and generally regarded as safe. Processes that use pressures between 100 and 400 bars and temperatures between 30 and 80 °C are feasible for the extraction of phenolic compounds, as presented in Table 5. In recent studies, under these conditions and depending on the natural source, extracts with TPC between 1.4 mg GAE/g and 113 mg/g extract have been obtained.

While $scCO_2$ is an excellent solvent of lipophilic compounds, when phenolic compounds are extracted using only $scCO_2$, the performance is relatively weak. However, is possible to improve the selectivity and yield of an extraction process using a co-solvent (Rosa et al. 2009). Ethanol is the most recommended co-solvent because it increases the solubility of the compounds. For example, ethanol has been employed to increase the extraction yield of phenolic compounds from *H. sabdariff* dried calyces (Pimentel-Moral et al. 2019) and feijoa peel (Henrique et al. 2019).

In SFE, the solvating power of the $scCO_2$–ethanol mixture depends on pressure and temperature. When pressure is increased, solvent density increases and higher extraction yields are obtained. In the extraction of phenolic compounds, pressures of up to 400 bars have been

Table 4 Summary of the recently works published on the extraction of phenolic compounds from natural matrices by SFE

Extraction conditions								
Matrix	Total phenolic content	Solvent	Temperature (°C)	Pressure (bar)	Extraction time (min)	S/F	Optimized parameters	References
Hibiscus sabdariff dried calyces	113 mg /g extract	$scCO_2$: ethanol 85:15–93:7	40–60	150–350	90	75	64 °C, 391 bar, $scCO_2$:ethanol 83:17	Pimentel-moral et al. (2019)
Horchata by-products	–	$scCO_2$	40	100–400	120	48	30 and 400 bar	Roselló-soto et al. (2019)
Arbutus unedo fruits	37.0 mg GAE/g d.w	$R_{S\text{-}L/CO2}$(%) 20–80	40–70	100–250	15	20	70 °C, 250 bar, $R_{S\text{-}L/CO2}$(%) 20	Alexandre et al. (2018)
Bio-oil from palm kernel shell	17.1 mg	$scCO_2$	50–70	300–400	60	–	70 °C, 400 bar	Chan et al. (2018)
Arctium lappa leaves	35.51 mg GAE/g extract	SMR (g $_{EtOH}$/g $_{CO2}$) 0–1.88	40–80	150–250	40–140	–	80 °C, 150 bar	Reder et al. (2018)
Garlic (Allium sativum L.)	–	$scCO_2$: ethanol 50:-50–70:30	30–50	100	13	–	50 °C, 100 bar, $scCO_2$:ethanol 70:30	Liu et al. (2018a, b)
Feijoa peel	23 mg GAE/g	$scCO_2$: ethanol 95:5	40–55	200–300	210	115	55 °C, 300 bar	Henrique et al. (2019)
Cacao pod husk	12.97 mg GAE/g extract	$scCO_2$: ethanol 95:5–85:15	40–60	100–300	150	180	60 °C, 299 bar, $scCO_2$:ethanol 86:14	Valadez-Carmona et al. (2018)

GAE, gallic acid equivalents; $scCO_2$, Supercritical CO2; $R_{S\text{-}L/CO2}$(%), volume ratio of solid–liquid/pressurized carbon dioxide; CP, compressed propane; SMR, initial ethanol to CO_2 mass ratio

Table 5 Summary of the recently works published on the extraction of phenolic compounds from natural matrices by UAE

Extraction conditions								
Matrix	Total phenolic content	Solvent	Temperature (°C)	Ultrasonic power (W)	Extraction time (min)	S/F	Optimized parameters	References
Lime peel waste	54 mg GAE/g d.b	Ethanol:water 50:50–100:0	–	Amplitude 20–40%	2–4	20	4 min, Amplitude 38%, Ethanol:water 55:45	Rodsamran and Sothornvit (2019)
A.satureioides inflorescences	37.3 mg QE/ml	Ethanol:water 0:100–100:0 Acetone:water 30:70–100:0 Methanol:water 30:70–100:0	25–50	100	10–90	20–60	25 °C, S/F 40, ethanol:water 70:30 or Acetone:water 50:50	Goltz et al. (2018)
Defatted oat bran	184.16 mg GAE/ 100 g d.b	Ethanol:water 80:20	20–90	600	25	10	70 °C	Chen et al. (2018)
Olive pomace	19.71 mg GAE/g d.b	Water	40	150–250	45–75	33–100	250 W, 75 min, S/F 50	Goldsmith et al. (2018)
Yellow soybean	495.37 μg/g d.b	Methanol:10% HCL 85:15 Acetone:10% HCL Ethanol:10% HCL 70% Methanol:70% Acetone 50:50 Acetone:water 50:50–80:20 Ethanol:water 80:20 Methanol:water 80:20	25	Amplitude 15–30%	2–15	20	10 min, Amplitude 30%, Methanol:10% HCL 85:15	Đurović et al. (2018)
Lemon waste	17.24 mg GAE/g	Water	23–50	150–250	10–60	100	40 °C or 50 °C	Papoutsis et al. (2018)
Olive leaves	37.44 mg GAE/g d.b	Water Ethanol:water 50:50 Methanol:water 50:50 Acetone:water 50:50	30–60	–	10–120	80	60 °C, Acetone:water 50:50	Irakli et al. (2018)

Table 5 continued

Extraction conditions								
Matrix	Total phenolic content	Solvent	Temperature (°C)	Ultrasonic power (W)	Extraction time (min)	S/F	Optimized parameters	References
Rheum moorcroftianum Rizomes	92.99 mg GAE/g d.b	Acetone:water 40:60	35–55	–	10–20	10–30	37 °C, S/F 28	Pandey et al. (2018)
Industrial potato by-products	–	Ethanol:water 100:0–50:50	35	190	10–45	20–90	35 min, S/F 10, Ethanol:water 55:45	Riciputi et al. (2018)
Morus alba L. leaves	15.29 mg/g	Deep eutectic solvents	40	600	30	20	Choline chloride/citric acid	Zhou et al. (2018)
Feijoa peel	68.2 mg GAE/g d.b	Ethanol:water 100:0–100:0	–	250	10	30	Ethanol:water 50:50	Henrique et al. (2019)
Rice grains	289.30 ± 0.12 µg/g	Methanol:water 100:0–50:50 pH 3–7	10–70	Duty cycle 0.2–0.7 s, Amplitude 30–70%	30	2.5–5	45 °C, 25 min, Duty cycle 0.4 s, Amplitude 47%, Methanol:water 80:20 (pH 4.25), S/F 1	Setyaningsih et al. (2019)
Mango peel	9.3 ± 0.4 mg GAE/g d.b	Ethanol:water 25:75–75:25	30	Amplitude 25–75%	20	25	Ethanol:water 50:50 without ultrasound	Guandalini et al. (2019)
Pomegranate peel	42.2 mg GAE/g	Water	–	Amplitude 20–100%	5–15	4	Amplitude 60%, 6.2 min	Sharayei et al. (2019)
Baobab (*Adansonia digitata*) seeds	4.1 mg GAE/g d.b	Methanol:water:HCL 80:19:1	40–60	Amplitude 30–50%	10–20	20–30	60 °C, 20 min, Amplitude 30%, S/F 30	Ismail et al. (2019)

GAE, gallic acid equivalents; QE, quercetin equivalents; CQAE, caffeoylquinic acid equivalent; LGH, lactic acid and glucose (5:1); CGH, citric acid and glucose (1:1); FCH, fructose and citric acid (1:1)

employed. For example, Roselló-Soto et al. (2019) observed a remarkable increase in the number and amount of phenolic compounds in SFE from horchata byproducts when the pressure was increased from 100 to 400 bars. A similar behavior was observed by Chan et al. (2018), who reported that the highest extraction yield of bio-oils from palm kernel shells was obtained at 70 °C and 40 MPa. Although SFE is recognized by its selectivity and the use of mildly temperatures, the main disadvantages of SFE are that polar compounds such as phenolics are difficult to extract without the use of co-solvents and the equipment necessary is more expensive.

Ultrasonic-assisted extraction

Ultrasound refers to frequencies from 20 kHz to 10 MHz. The food industry uses two types of frequencies: diagnostic ultrasound (2–10 MHz) and conventional power ultrasound (20–100 kHz) (Pingret et al. 2013). Diagnostic ultrasound is used in medical imaging and defect detection, whereas conventional power ultrasound is used in the extraction of bioactive compounds, defoaming, degassing, and sterilization, among other applications. In this process, cavitation induces a series of compressions and rarefactions in the liquid medium that causes pressure changes and the formation and collapse of bubbles (Tiwari 2015). The implosion of gas bubbles in a liquid medium generates a rapid change of heating up to 5500 °C and a pressure increase to 500 bars. During cavitation, the cells walls of the matrix are disrupted, allowing the extraction solvent to penetrate deeply and enhancing mass transfer. UAE is associated with greater extraction yields and faster extraction rates and uses lower temperatures and smaller quantities of solvents (Rutkowska et al. 2017).

The simplest UAE equipment is the ultrasonic cleaning bath. This device is easy to handle and has a low implementation cost. On the other hand, the probe system is more adaptable and is considered more powerful than cleaning baths, as there is less dispersion of ultrasonic energy (Pingret et al. 2013). UAE systems are composed of a power generator, transducer, amplifier, and probe. The transducer converts electrical energy into acoustic energy by vibrating mechanically at ultrasonic frequencies. The raw material is placed in an extraction vessel, then the solvent at the desired temperature is added and the ultrasonic process begins. After extraction time, the extract is recovered after filtration.

Ultrasound power and amplitude are among the most important process parameters evaluated in the UAE of phenolic compounds (Table 5). Ultrasonic power or intensity is therefore proportional to amplitude. Ultrasonic power and amplitude have a positive relationship with the extraction of phenolic compounds. According to Rutkowska et al. (2017), the highest yields in UAE are usually achieved by increasing ultrasound power. Goldsmith et al. (2018), who studied the extraction of phenolic compounds from olive pomace by UAE, obtained an increase in TPC when ultrasound power was increased from 150 to 250 W. In that study, UAE yielded a higher level of TPC as well as antioxidant activity compared with LPSE. This behavior can be explained by the fact that when ultrasonic power increases, major alterations in the plant matrix can be caused by cavitation. Solvent penetration is therefore enhanced and more phenolic compounds are recovered. Consequently, ultrasound power and amplitude are crucial parameters that should be optimized to maximize yields and minimize energy consumption. Variation in ultrasound power and amplitude can result in a certain selectivity of target molecules, in which the ratio of some molecules is a function of the applied power (Pingret et al. 2013). Đurović et al. (2018) studied the extraction of phenolic acids from soybean seeds by UAE followed by alkaline and acid hydrolysis. In their study, the total content of phenolic acids increased when the amplitude was increased from 15 to 30%. However, although some phenolic acids such as trans-cinnamic or caffeic acid can be released by UAE, compounds such as p-coumaric and ferulic acids are not released despite increased amplitude. These compounds are strongly joined to soybean cell components and would be require more ultrasonic power to release them.

UAE is one of the most studied techniques for the extraction of phenolic compounds due to its performance, short extraction time, and use of mid-range temperatures. According to data gathered in the literature survey, extraction of phenolic compounds is performed using extraction times and temperatures of between 2 and 120 min and 20 and 90 °C, respectively. As with the other extraction methods, an increase in temperature enhances the solubility and diffusion of phenolic compounds. In UAE of phenolic compounds from defatted oat bran (Chen et al. 2018), TPC increased when the temperature was increased from 20 to 70 °C and TPC obtained at 70 °C was almost two times higher than that obtained at 20 °C after 5 min of UAE. Similar behavior also was observed by Papoutsis et al. (2018), Irakli et al. (2018), Riciputi et al. (2018), and Pandey et al. (2018) in the extraction of phenolic compounds by UAE from citrus pomace, olive leaves, potato byproducts and *Rheum moorcroftianum*, respectively. However, although the increased temperature enhances mass transfer and extraction yields are improved, bioactive compounds can be extremely heat sensitive and lose their antioxidant activity when the temperature is raised. Such behaviors were observed by Goltz et al. (2018) during the extraction of phenolic compound from *A. satureioides* inflorescences, in which TPC was negatively

influenced when temperature was increased from 25 to 50 °C.

In general, lower temperatures and lower ultrasound power or amplitude results in lower phenolic extraction yields. When the value of these process parameters is raised, the phenolic extraction yield increases up to a certain value, after which the phenolic compounds are degraded and extraction yields falls. Therefore, although the phenolic content depends on the raw material source, extraction efficiency is related to the optimization of the process parameters.

Comparisons between conventional and alternative extraction methods

Both conventional and alternative extraction methods use polar solvents. The solvent plays a crucial role in the extraction of phenolic compounds and organic solvents of higher polarity are more useful than nonpolar solvents in the extraction of phenolic compounds (Oreopoulou 2003). Currently, GRAS solvents such as water and ethanol, are preferred. The ethanol–water mixture is more effective in the extraction of phenolic compounds than single solvents. This can be explained by the intermediate polarity of hydroalcoholic mixtures, similar to the phenolic compounds which increase the solubility of the target compounds. In the case of the phenolic compounds, its solubility is enhanced by ethanol, whereas water enhances its desorption from the sample.

Conventional extraction methods have two major drawbacks when compared with the alternative methods: use of high temperatures and longer extraction times, both of which can trigger degradation of phenolic compounds. In Soxhlet extraction, the mixture of solvent and extract is heated continuously to its boiling point using longer extraction times. The extracts obtained using this technique, therefore have fewer phenolic compounds. When Alara et al. (2018a, b, c) compared MAE and Soxhlet extraction of phenolic compounds from *V. amygdalina* leaves, MAE produced a higher extraction yield in a shorter time. MAE obtained a TPC of 102.04 mg GAE/g d.b. in 10 min, whereas Soxhlet extraction obtained a TPC of 73.54 mg GAE/g d.w in 480 min.

Regarding extraction time, conventional methods are more time-consuming when compared with alternative processes. For instance, Soxhlet and maceration extraction of phenolic compounds from feijoa peel (Henrique et al. 2019) and hazelnut shells (Yuan et al. 2018a, b) used extraction times of 6 and 12 h, respectively. Although maceration did not use the elevated temperatures of the Soxhlet extraction, its longer extraction time proved to be its main disadvantage. Ismail et al. (2019) carried out a comparative study between UAE and maceration in the extraction of phenolic compounds from baobab seeds. The results indicated that UAE resulted in a significant higher TPC (418.01 mg GA/100 g d.b.) compared with maceration (357.34 mg GA/100 g d.b.). Maceration had a lower TPC than UAE, and it involved an extraction time of 24 h whereas UAE required only 20 min, which represents an extraction time 72 times longer.

Despite these disadvantages, conventional methods remain in use because the extraction units are widely available and are less expensive than those for MAE, PLE, SFE and UAE alternatives.

Comparison among the alternative extraction methods

Several studies of extraction of phenolic compounds have been performed using alternative methods. Process parameters such as extraction time, solvent choice, pressure, temperature, microwave power, and ultrasound power are important determinants of process performance. Most of these parameters can have individual or combined effects on the performance.

Extraction time

Extraction time is a key factor that determines energy consumption and process feasibility. Combined with temperature, it plays a critical role in compound stability. It is generally possible to observe that extraction processes that do not need to pack the raw material into an extraction vessel are faster. For example, among the alternative processes, MAE and UAE use shorter extraction times than PLE and SFE. In MAE extraction times as brief as 0.75 and 1.15 min have been found in the optimization of extraction of phenolic compounds from lime peel waste (Rodsamran and Sothornvit 2019) and *Scirpus holoschoenus* rhizomes (Oussaid et al. 2018), respectively. In the same way, extraction times of up to 4 min have been used in the extraction of phenolic compounds from lime peel waste (Rodsamran and Sothornvit 2019). However, although PLE also involves shorter extraction times, such as 5 min (Lee et al. 2018), extraction times as long as 180 or even 480 min are associated with the extraction of phenolic compounds from *Hancornia speciosa* (Barbosa et al. 2019) and pistachio hulls (Erşan et al. 2018), respectively. Although shorter extraction times, such as 13 min can be seen with SFE, the average extraction time is above 60 min. In fact, SFE required the longest extraction times, which is the main drawback of this extraction process.

Temperature

MAE and PLE require the highest temperatures. Although higher temperatures enhance the release of greater amounts of phenolic compounds, the temperature must be limited due to thermal instability of the compounds. The use of high temperatures and pressures is associated with degradation of phenolic compounds. For example, in the extraction of phenolic compounds from *H. sabdariffa* by MAE, Pimentel-Moral et al. (2018) observed that some thermo-labile compounds were degraded in temperatures above 100 °C. PLE uses the highest temperatures. For example, in the extraction of phenolic compounds from Pacific oysters (Lee et al. 2018), *Stevia rebaudiana* (Bursa et al. 2018), and barley (Huerta and Saldaña 2018), temperatures between 160 and 225 °C have been used. The use of high temperatures in PLE triggers degradation of the compounds. Okiyama et al. (2018) observed a drop in total flavanol content at temperatures of 75 to 90 °C after 40 min of extraction. A similar behavior was observed by Erşan et al. (2018) in the extraction of phenolic compounds from pistachio hulls. In that study, when the extraction temperature was raised from 110 to 150 °C, the phenolic yield was increased. However, increasing the temperature from 170 to 190 °C resulted in a significant decline in phenolic yield.

To preserve the integrity of the compounds, all alternative extraction processes should use mid-range temperatures. SFE uses a lower average temperature (40 °C). This can be explaining by the properties of the supercritical CO_2–ethanol mixture, which is the most common solvent used in extractions. SFE allows for the extraction of phenolic compounds using moderate temperatures and the compounds can be recovered easily from the supercritical fluid by reducing pressure. However, due to the polarity of the phenolic compounds, a co-solvent is generally needed.

Solvent

As previously mentioned, organic solvents of higher polarity are more efficient than nonpolar solvents. In SFE, a polar solvent is used as a co-solvent to extract phenolic compounds because when scCO2 is used alone, the extraction yield of phenolic compounds is poor. For example, in the extraction of phenolic compounds from *H. sabdariff*, Pimentel-Moral et al. (2018) reported that the extraction of these compounds increased when larger amounts of co-solvent were used.

In some cases, however, the use of acidified solvents increases process performance. The extraction yield of phenolic compounds by PLE and UAE from grapes (Vitor et al. 2019) and yellow soybeans (Đurović et al. 2018) in acid mediums increased. The low pH contributed to cell wall disruption and enhances mass transfers (Vitor et al. 2019). In addition, the use of green and sustainable solvents known as deep eutectic solvents (DESs) has attracted much attention. DESs are systems normally prepared from non-ionic starting materials, such as molecular compounds and salts. For example, Zhou et al. (2018) evaluated the effect of DESs (including choline chloride-, betaine-, and L-proline-based solvents) on the extraction of phenolic compounds from mulberry leaves by UAE. In that study, a phenolic content of 22.66 mg/g was obtained when a solvent composed of choline chloride/citric acid was used, whereas a phenolic content of 15.29 mg/g was obtained with methanol. DESs are therefore associated with more effective extraction yields.

Yield and efficiency

In addition to process parameters, extraction yield and phenolic content also depend strongly on the raw material, cultivar, and ripening stage. Therefore, it is difficult to conduct comparisons of the performance of alternative extraction techniques based on extraction yield and phenolic content. All alternative extraction techniques produce high TPC results. A literature search revealed that MAE produces the highest TPC, at 227.63 mg GAE/g d.b. (Nabet et al. 2019), followed by PLE, UAE and SFE, with 173.65 (Otero et al. 2019), 92.99 (Pandey et al. 2018) and 37 mg GAE/g d.w (Alexandre et al. 2018), respectively. As expected, the lowest TPC was obtained when SFE was used. Although a co-solvent be used, SFE is an effective technique for the extraction of essential oils and nonpolar compounds, and the performance of the MAE, PLE and UAE is better.

In some studies, the same raw material is extracted using two or more extraction techniques to compare different extraction methods. For example, Rodsamran and Sothornvit (2019) compare the efficiency of MAE and UAE. In that study, UAE exhibited superior performance to extract total phenolics compared with MAE. Under optimal extraction conditions, UAE had the highest efficiency, obtaining a TPC of 54.4 mg GAE/g, a time saving of 33% compared with MAE. Pomegranate peels have been used to obtain phenolic compounds using MAE (Kaderides et al. 2019) and UAE (Sharayei et al. 2019). A TPC of 199.4 mg GAE/g using an extraction time of 4 min was obtained using MAE whereas a TPC of 42.2 mg GAE/g was obtained with UAE and an extraction time of 6.2 min. Although in this case the performance of MAE surpassed that of UAE, these contradictory results can be explained by the fact that the performance of the extraction process is influenced by the interaction of several factors. Therefore, selection of the proper extraction process should consider

extraction parameters as well the characteristics of the raw material.

Process combination and integration

Extraction yield and recovery of phenolic compounds can be enhanced by combining and integrating alternative methods. Among the different possible combinations, ultrasound combined with other alternatives appears to be a promising option. Ultrasound enhances mass transfer and breaks up the cellular matrix, releasing greater quantities of target compounds and producing larger extraction yields. The combination of ultrasound and PLE was studied by Sumere et al. (2018) for the extraction of phenolic compounds from pomegranate peels. In that study, extraction was improved by combining these two techniques. Ultrasound enhanced extraction yields mainly when large particles were used and the temperature and ultrasound power ranged between 70 and 80 °C and 480 and 640 W, respectively. In this case, UAE combined with PLE allowed for the use of water as the extraction solvent while reducing extraction time. The combination of ultrasound and SFE has also been studied. Santos-Zea et al. (2019) evaluated the effect of ultrasound on SFE in the recovery of antioxidants from agave bagasse. In that study, the recovery yield of antioxidants increased when a multiplate ultrasound transducer was used. The use of the ultrasound combined with SFE resulted in 1.7-fold and threefold increases in extraction of antioxidants and saponins, respectively, and showed that transducer geometry can significantly enhance the intensification effect of ultrasound in SFE processes.

Integration of alternative extraction processes is an attractive approach to producing different valuable products of higher quality from the same matrix, e.g., essential oils (nonpolar fraction) and phenolic compounds (polar fraction). These kinds of integrated processes present promising alternatives not only for obtaining phenolic compounds but for extracting other bioactive compounds from natural sources. For example, de Aguiar et al. (2019) proposed an integrated extraction process of bioactives from biquinho peppers. First, SFE was used to extract the nonpolar fraction and then a PLE was performed to recover the phenolic compounds from the SFE-extracted biquinho pepper. The researchers obtained a capsiate-rich oleoresin (8.67 mg/g) from the SFE and an extract with up to 16 phenolic compounds (hydroxyben-zoic and hydroxycinnamic acids, flavonoids and glycosides).

Concluding remarks

Industry and consumers are aware that compounds derived from natural sources can prevent and treat certain illnesses. Phenolic compounds are a promising option for the development of products for both the food and pharmaceutical industries. A key factor in all cases is the extraction process. However, extraction can be challenging because of the instability of the desired compounds. A useful extraction process therefore preserves the integrity of the compounds. Alternative process such as MAE, PLE, SFE, and UAE offer an effective alternative process for obtaining extracts rich in phenolic compounds that preserves bioactivity. These processes allow for the extraction of phenolic compounds using lower solvent consumption rates, shorter extraction times, and solvents that are generally as safe. For MAE, PLE, and UAE, the use of an ethanol–water mixture is recommended. For SFE, the use of ethanol as a co-solvent is the best option. MAE and PLE use the highest temperatures. Although the temperature should be limited due to the thermal instability of the compounds, a degradation temperature should be determined for each raw material and extraction process. Among alternative extraction techniques, MAE could be the most promising process because it is associated with the highest TPC using shorter extraction times, followed by UAE, PLE, and SFE.

Extraction performance can be improved by combining or integrating two different extraction techniques. Combining ultrasound with PLE or SFE enhances extraction efficiency. In the future, process integration will allow the whole use of raw materials in a process that produces the nonpolar and polar fractions separately. In this case, the nonpolar fraction would be obtained by SFE and afterward, the phenolic compounds would be obtained by MAE, UAE, or PLE. The extraction process should guarantee the bioactivity of the compounds and ensure all solvents are recycled.

Since plants are an excellent source of phenolic compounds, it could be turned into a real source of natural products to substitute synthetic food additives in the near future. At present, the use of alternative extraction techniques and its scale-up should start to develop processes at the industrial scale to obtain more food ingredients based on phenolic compounds with future applications in the market. Further research and greater effort are necessary to develop this kind of processes to the industrial scale.

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