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Effect of Extraction Methods on Bioactive Compounds of Plant Origin

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ABSTRACT

The use of bioactive compounds has been maintaining its significance from nutritional aspects. Due to the increasing demand for them in potential markets, researchers struggle to create new sources and improve their methods. Plant materials possess plenty and a diverse range of these compounds. However, their availability strongly depends on the extraction techniques in addition to the sampling methods and the applicability of the method to the specific parts of the plant. Thus, it is crucial to develop a common, precise way which will enable to extract all the active components regardless of their origin and their location in the plant material. Besides, the new method ought to have the highest economic value in comparison to the present applications which means that the efficiency of the extraction should be acceptable on industrial scale as well. Even though numerous methods have been improved so far, it seems to be unlikely to achieve a standardized solution with high valorization for the extraction of bioactive compounds from plants until now. This review aims to discuss the novel extraction methods in addition to the conventional techniques focusing on the critical parameters such as the cost, time, yield, feasibility and eco-friendliness of the process.

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Introduction

Today's world society strongly intends to consume both foods useful for the metabolic functions of human body and other chemical compounds that will have beneficiary effects on our health as well. These compounds categorized as non-nutritional ingredients are called bioactive compunds. Bioactive compounds (BA) are secondary metabolites produced by the organism to arrange physiological and cellular activities and to increase their resistance to survive (Harborne, 1982).

Several research in the past reveal that BA serve as health promoters when they function as cofactor or inhibitor in enzymatic reactions, as substrate in biochemical reactions, as absorbent for the removal of undesirable compounds in the gastrointestinal tract, as fermentation substrate for the useful microorganisms, as inhibitors preventing from the growth of harmful bacteria and as scavenging agents for the reactive and toxic chemicals (Kris-Etherton et al., 2002).

Extraction is the critical stage of food processing with high added value since health promoters such as BA must be obtained adequately and damage-freely from the raw material. Thus, the choice of the most convenient extraction method specific to each bioactive compound plays an important role. This review intends to specify possibly alternative technologies for the extraction of a diverse range of BA in terms of economy and environmental concern on industrial scale.

Extraction Methods

The description, identification and classification of BA can only be performed if certain extraction methods are applicable to the nature of the source with respect to the selectivity of BA under different conditions (Azmir et al., 2013). The common objectives of the extraction methods can be summarized as

- To extract the analyte from the complex matrix
- To increase the selectivity and sensitivity of the method
- To convert these compounds into a simpler form providing a quantitative and qualitative analysis
- To enhance the reproducibility of the study regardless of the variables related to the sample composition (Smith, 2003).

Conventional Extraction Methods

Conventional extraction methods are based on the solvation power, agitation process and heat stability of analyte. The methods can be classified into three groups namely as Soxhlet extraction, Solvent extraction, Hydrodistillation.

Soxhlet extraction: This method has been widely used in recent studies for the extraction of BA, although it was first developed in 1879 by Franz Ritter Von Soxhlet in order to extract lipids from plant materials. It can be used as an alternative to the novel extraction techniques to make a comparison between the methods. The principle

of Soxhlet extraction (SOE) implies the frequent treatment of fresh solvent with the sample after each reflux cycle of the solvent followed by evaporation at high temperatures and condensation of the solvent (Castro and Capote, 2010). This is one of the most favourable features of this method since the solvent with extremely high purity evaporates and then condenses to extract the solid sample in each cycle. Therefore, the extraction yield of SOE is higher than the yield obtained by conventional solid liquid extraction. At the end of the process, the solvent might be easily vaporized by a rotary evaporator to obtain the plant oil. The other benefits of SOE can be arranged as follows:

- No need for a filtration process
- Extraction temperature and the heat transfer rate by the distillation vessel is high.

On the other hand, several drawbacks of SOE can be listed as follows:

- Excess of solvent consumption
- Degradation of heat labile analytes
- Long extraction time
- Lack of mixing process
- Difficulty in evaporation of vast amounts of solvents
- (Wang and Weller, 2006).

Solvent extraction: Solid liquid extraction (SLE) is the method relying on the migration of solid particles into the liquid by diffusion and mass transfer principles after the treatment with the liquid solvent. When a solvent is added into the food samples and then the mixture is shaken thoroughly, the solid particles swell by sorbtion and the dissolved particles migrate slowly into the solvent via capillary and diffusion effect (Self, 2005). The mass transfer might be increased by altering the boundary layer, concentration gradient and coefficient of diffusivity (Corrales et al., 2009). Extraction yield is a function of process conditions and depends upon temperature, solid to liquid ratio (S/L), particle size and the concentration of the bioactive compound which is desired to be extracted from the plant. Besides, extraction time and S/L are considered as signinificant process parameters influencing on the quantity of phenolic compounds (PC) to be extracted (Hayouni et al., 2007; Pinelo et al., 2004; Rubilar et al., 2003). Moreover, the type of solvent varies with regard to the type of the bioactive compound. The most extensively used solvents are reported as acidified methanol or ethanol (Amr and Al-Tamimi, 2007; Awika et al., 2004; Caridi et al., 2007; Lapornik et al., 2005). The polarity of the target analyte is one the most critical factors in the selection of the proper solvent. Meanwhile, the solvent with the highest extraction yield has been pointed out as methanol (Kapasakalidis et al., 2006). However, due to the toxicity of methanol, it is avoided to use in food industry and thus ethanol is more preferable for the extraction process. The types of solvents used in the extraction of BA are listed in Table 1.

During the extraction, PC are seperated in a systematic and sequential order. Phenolic acids are present in the food matrix in both free and bound forms. The extraction of free phenolics (phenolic acids, soluble esters, soluble glycosides) takes place in the aqueous mixtures of organic solvents (Escarpa et al., 2002; Mattila and Kumpulainen, 2002; Russell et al., 2008). Phenolic acids may also form complexes linked to the cell wall by insoluble esters and glucosides. These structures may turn into the free form if only alkaline, acid or both of alkaline and acid hydrolysis occurs (Mattila and Kumpulainen, 2002).

Liquid-liquid extraction (LLE) is the method which includes the treatment of a liquid that can dissolve one or more specific compounds with an almost immiscible solvent. Hence, the selectivity of the liquid solvent owing to the density difference is crucial. At the end of extraction two distinct phases are observed. The first phase called extract contains the desired compounds whereas the second phase called raffinate is composed of the residue with a lower concentration of the target compounds. This method is usually feasible for the extraction of BA from liquid by products in beverage industry (Ignat et al., 2011). Its drawbacks can be underlined as using expensive and toxic solvents in addition to the involvement of a couple of stages required for the elimination of non-phenolic compounds (terpenes, chlorophyl, waxes) with high operation costs (Naczk and Shahidi, 2006; Gomez et al., 2005). Extraction efficiency and degradation degree of phenolic substances significantly depend on temperature, presence of light and air and several process variables such as agitation time, centrifuging period, evaporation time (Salas et al., 2010).

Hydrodistillation: Hydrodistillation (HD) is the traditional extraction process of BA and essential oils (EO). It may appear in three different forms in the industrial applications: a) Distillation by distilled water b) Water and water vapor distillation c) Direct water vapor distillation (Vankar, 2004). Within the framework of this method, the sample material is installed into a closed vessel. Then, distilled water is added adequately into this vessel and the mixture is allowed to boil. Alternatively, steam injection can be performed in this stage. Hot water and steam help to liberate BA from plant tissue. The indirect cooling by water allows water-vapor-oil mixture to condense and this mixture flows automatically from condenser into the separator where oil and BA are separated from water (Silva et al., 2005). HD involves three steps called hydrodiffusion, hydrodistillation and hydrolysis by heat. Therefore, some volatile compounds might be lost at high extraction temperatures (Azmir et al., 2013).

The earlier studies conducted so far in order to extract BA from several plants by traditional methods are summarized in Table 2.

Novel Extraction Methods

In order to eliminate the drawbacks of traditional extraction methods such as long extraction periods, necessity of using solvents with high purity, low extraction selectivity, solvent consumption in huge quantities and degradation of heat labile components, new methods have been implied (Luque de Castro and Garcia-Ayuso, 1998). Novel techniques are described as Ultrasound assisted extraction (UAE), microwave assisted extraction (MAE), accelerated solvent extraction (ASE), pulsed electrical field extraction (PEF), enzyme assisted extraction (EAE), supercritical fluid extraction (SFE) and high hydrostatic pressure extraction (HHP).

Table 1 Solvents used for the extraction of phenolic compounds from plant materials

Phenolic compound	Solvent	Reference
Phenolic acids, flavonol, anthocyanin	Ethyl acetate	Pinelo et al., (2004); Russell et al. (2008)
Anthocyanin, catechin, phenolic acids, flavonon, flavonol, flavon, procyanidin, ellagic acid, rutin, chlorogenic acid	Aqueous mixtures of methanol with different volume ratios (50-90% v/v)	Bleve et al., (2008); Caridi et al. (2007); Ross et al. (2009); Mattila and Kumpulainen (2002)
Anthocyanin, flavonols, free phenolic acids	Aqueous mixtures of ethanol with different volume ratios (10-90% v/v)	Balas and Popa (2007); Wang et al. (2009); Bleve et al. (2008), Bucic-Kojic et al., (2011); Corrales et al. (2009); Ross et al. (2009)
Flavonol, Free phenolic acids	Chloroform	Sharififar, Dehghn-Nudeh and Mirtajaldini (2009)
Flavonols, phenolic acids	Diethyl ether	Ross et al. (2009)
Proanthocyanidins, phenolic acids	Hot water (80-100°C)	Diouf et al. (2009)
Flavonols, phenolic acids, hydroxy cinnamic acid, cumarin, flavonol, ksanton	Aqueous mixtures of acetone with different volume ratios (10-90% v/v)	Naczk and Shahidi (2006); Sharififar et al. (2008); Schieber et al. (2003)
Tannins and bound phenolic acids	NaOH (2-10 M)	Nardini et al. (2002); Popa et al. (2008); Ross et al. (2009)
Oleoropein, rutin and other polyphenols (olive leaves)	Aqueous mixtures of Acetone and ethanol at different ratios (10-90% v/v)	Altıok et al. (2008)

Table 2 Studies focusing on the extraction of bioactive compunds from plant materials by conventional methods

Method	Analyte	Plant	Conditions	Result	Reference
SLE-Alkaline hydrolysis	Phenolic acids	Vine yards	Treatment with 3% H_2SO_4 at 130^0 C for 15 min. Treatment with 4-12% NaOH (w/w) for 30-120 min. at 50-130^0C	Ferulic acid: 25.7-141 mg/L p-coumaric acid: 15.5-31.5 mg/L Gallic acid: 2.5-164.6 mg/L	Max et al. (2010)
SLE- Acid + Alkaline hydrolysis	Phenolic acids liberated from soluble esters and soluble glycosides	Wheat, rye, triticale	Mixing with 80% methanol at 80^0 C for 15 min.; addition of 6 M HCl to pH=2, dissolving the suspension in 2 M NaoH under N_2; isolation of free phenolics by diethyl ether at 100^0 C for 1 h under N_2	Caffeic, p-coumaric, ferulic and sinapic acids were detected by HPLC Majoirty of free phenolics is in form of soluble esters	Weidner et al. (1999)
SLE, SOE	Flavonoids	Corn	SLE: Methanol (60% v/v) for 24 h in the dark SOE: 95 ^{0}C-30 min. with Methanol (60% v/v)	Total flavonoid recovery SLE (%95); SOE (%80)	Biesaga (2011)
SOE	Isoflavones	Soybean	Boiling with dimethyl sulphoxide: acetonitrile: water (5:58:37, v/v/v) for 3 h then rinsing the residue twice with the same solution	SOE recoveries are 73.2% as compared to UAE and 68.3% as compared to ASE.	Luthria et al. (2007)
HD	Essential oils	Pennyroyal leaves	50 g of sample was extracted in Clevenger apparatus for 3 h	Extraction recoveries of oil were reported for particle sizes of 0.7, 0.5 and 0.3 mm as 2.74%; 2.49% and 2.11% respectively	Reis-Vasco et al. (1999)
SLE- Acid Hydrolysis	Flavonoids	Herbs: *Rosa damascena*, *Solidago virgaurea*, *Ginkgo biloba*, *Camellia sinensis*	50% Methanol + 1.2 M HCl at 80^0 C for 2 h	Quercetin: 0.54-11.10 mg/g; kaempferol: 0.03 - 14.80 mg/g; isorhamnetin: 0.19-2.76 mg/g; luteolin: 0.15 - 2.36 mg/g; Apigenin: 0.27-2.05 mg/g; Myricetin:0.42-1.82 mg/g in dry plant samples	Haghi and Hatami (2010)

Ultrasound assisted extraction: Ultrasound waves are certain types of electromagnetic radiation which propagate through a medium with a frequency range between 20 kHz and 100 MHz (beyond human hearing) by generating compression and expansion. (Chemat et al., 2011). The basic mechanism of an ultrasound assisted extraction (UAE) device is composed of several components: a) The ultrasonic electric generator which creates a signal (usually around 20 kHz) that powers the transducer, b) The transducer whose function is to convert a specific type of energy into another form. This part of the device seems to a prob where electrical energy is converted into ultrasound energy or vice versa. Using piezzoelectric crystals the transducer converts the electrical energy into mechanical vibrations. c) The sonicator which enables to amplify these vibrations until they pass through to the probe, d) the probe whose function involves in transmission of the vibration to the solution being sonicated.

The energy conversion in the ultrasound mechanism takes place in the following manner: First high voltage of electric energy and current is applied to transducer and this energy is translated into mechanical energy. Then, the transducer generates acoustic waves and subsequently the cavitation bubbles are formed. The cavitation is defined as the formation of the bubbles inside the liquid and the collapse of these bubbles following this event (Kındır, 2010). The implosion of cavitation bubbles leads to the generation of extreme temperatures (5000 K) and pressures (1000 atm), which produce, in turn, substantially high shear energy waves and turbulence in the cavitation zone (Soria and Villamiel, 2010).

Several process parameters such as temperature, viscosity, amplitude, time of exposure are involved in UAE. When temperature is increased, the viscocity of the sample falls down, the number of the cavitation bubbles rise since they are formed more easily in a less viscous environment. There is an optimum temperature at which the viscosity is low enough to form adequate cavitation bubbles. On the other hand, the temperature must be maintained at such a level so that no dampening may occur due to high vapor pressure (Patist and Bates, 2008). The energy intensity is directly related to the amplitude which depends on the power input of the transducer. At higher intensities, the growth of smaller bubbles is also accelerated. When the external pressure is risen, faster or more violent collapse of the bubbles are formed. The time of exposure is associated with the flow rate of the sample into the ultrasonic device. The higher the energy input, the lower the flow rate (Patist and Bates, 2008).

The ultrasound frequency is also one of the critical process parameters in the extraction. Vinatoru (2001) reported that, although the extraction yield did not alter significantly, the higher the frequency the lower the degradation of the herb which might be helpful during the extraction of toxic alkaloids.

The major advantages of sonication can be counted as shorter extraction time, energy and solvent savings (Azmir et al., 2013). Furthermore, a more rapid agitation which facilitates the mass transfer, use of instruments with small volume and faster energy transfer provide an effective control of process parameters (Chemat et al., 2008). Soria and Villamiel (2010) emphasized that, UAE was as effective as any other high temperature longtime extraction process because it could greatly decrease the extraction time. The efficiency of UAE could be associated with the simultaneously enhanced hydration and fragmentation process in addition to the facilitation of the mass transfer of solutes to the extraction solvent.

UAE can be performed using two different types of equipment called sonicator and ultrasound (sonication) bath. The former leads to frequent pollution and thus has lower reproducibility when the tips which densely come into contact with sample surface are not meticulously clarified after each process. Cleaning process of the tips attached to the probe might be exhausting and time consuming due to the physical nature and roughness of these tools. On the other hand, the latter is able to overcome this problem and facilitates the extraction of a number of samples simultaneously. Besides, ultrasonic baths can be installed and operated at lower costs and they might also be used for multifunctional purposes such as degassing and cleaning of the glassware. However, the ultrasonic probe system is considerably more effective in the extraction due to the high intensity caused by the contact on a much smaller sample surface (Chemat et al., 2011). A higher extraction yield of some essential oils from herbs might be gained in the former case. Vinatoru (2001) reported substantially higher yields of cineole, thujone, borneol from sage by the probe application in comparison with ultrasonic bath and especially in a shorter time. On the other hand, the rapid increase in temperature of the sample particularly while working with small volumes of samples might be regarded as a drawback of the probe system (Chemat et al., 2011).

Several reports in literature point out that better yields and shorter extraction time might be achieved by UAE compared to conventional techniques. Extraction of diverse aroma compounds from tea (Xia et al., 2006) wine (Cabredo-Pinillos et al., 2006) and aged brandies (Caldeira et al., 2004) by using ultrasonic baths was reported. In contrast to the traditional methods, operation at low temperatures prevents from higher thermal degradation of some essential oils extracted from several spices such as artemisia (Aswaf et al, 2005), garlic (Kimbaris et al., 2006), lavender (Porto et al., 2009), peppermint leaves (Shotipruk et al., 2001). Zu et al. (2012) tested different ionic liquids with UAE to extract carnosic acid and rosemarinic acid from plant tissue and performed an optimization by response surface methodology. As a result, it was reported that the extraction efficiency by 80% ethanol using ultrasonic device is quite similar to conventional extraction (100% for carnosic acid and 64.9% for rosemarinic acid) and the extraction time is considerably shorter (30 minutes) than those yielded by traditional solvent extraction (96% efficiency for carnosic acid, 67.3% for rosemarinic acid in 24 hours).

Microwave assisted extraction: Microwaves are type of electromagnetic radiation composed of two oscillating fields such as electric and magnetic field perpendicular to each other. They include a frequency range between 300 MHz and 3000 GHz. MAE is based on the conversion of electromagnetic energy into heat energy by dipole rotation and ionic conduction mechanisms (Jain, 2009). Primarily, heat is generated thanks to the resistance of the

sample to ionic flow within the conduction mechanism. Then, the ions change their direction as the sign of the field changes. During the permanent change of the directions collisions take place between molecules.

MAE is considered to involve three serial steps revelaed by Alupului (2012): a) Under elevated temperature and pressure the soluble substances are seperated from the active side of the sample; b) Penetration of the solvent across the sample is facilitated; c) Transition of soluble compounds into the solvent occurs. Several process variables such as microwave power, frequency, the moisture content of food, particle size, concentration and type of solvent, solid to liquid ratio, number of cycles, temperature and pressure are supposed to influence the extraction yield of secondary metabolites (Wang and Weller, 2006). MAE is a technique that provides the alternative to the user to choose working with or without solvent. In case of using a solvent, dielectric constant, dissipation factor, boiling point and polarity of the solvent and sample must be taken into consideration since the performance of the extraction depends on the conformity of these properties (Khoddami et al., 2013).

MAE can be carried out in five different ways with respect to the compatibility of the controllable process variables with the properties of the sample and analyte: a) MAE under vacuum (VMAE); b) Nitrogen protected MAE (NMAE), c) MAE operating in dynamic mode (DMAE); d) Solvent free MAE (SMAE) e) Combination of UAE with MAE (UMAE) (Chan et al., 2011). VMAE, is operating up to 1000 W for short periods (2-20 min.) and especially favourable in contrast to other MAE techniques when the sample is more likely to be exposed to deterioration by heat and air. If, a food sample with a substantial heat resistance is to be analyzed, NMAE is preferable. When time and efficiency of extraction are considered as the primary factors for industrial applications, DMAE might be a better choice. For the extraction of EO, SMAE can be suggested as an appropriate method. In order to intensify the field effect for a wide variety of samples with different chemical nature, UMAE might be chosen as well (Chan et al., 2011).

Pulsed electric field extraction: Pulsed electric field extraction (PEF) is the process in which the food sample is placed between two electrodes and electric current is allowed to pass along a period of 1-100 µs producing a field intensity varying from 1 to 80 kV/cm (Seçkin and Özgören, 2011). Within a short duration of intense electric current, a considerable change in pressure along the cell wall can be ensured. When the sample is exposed to the current exceeding a certain critical value, the membrane of the cell wall is torn which enables the permeability. The continious process results in pore formation which is defined as electroporation (Singh and Yousef, 2001; Pizzichemi 2007).

Unfortunately, the reason for such a pore formation is quite ambiguous. When the electric field intensity, the number of pulses and the pulse width are increased, the size of the pores also rise and the pore formation becomes irreversible. The consequent pores bring about the disruption of the cell tissue (Zderic and Zondervan, 2016).

The effectiveness of PEF application may be generally associated with the electric field intensity, number of pulses, energy density per unit mass of sample, temperature, duration of the treatment, the structure of the food matrix. (Heinz et al., 2003)

It is proposed that PEF seems to be less time consuming and to have less detrimental effects on the chemical, physical and sensory characteristics of the food matrix in contrast to the other extraction techniques. On the other hand, its use has been restricted by the liquid samples with specific electrical conductivity (Han, 2007).

Enzyme assisted extraction: The basic principle of enzyme assisted extraction (EAE) comprises the disruption of hydrophilic and hydrophobic linkages between phenolic compounds and cell wall by the addition of enzymes (Pinelo and Meyer, 2008). Some phytochemicals exist in plant tissue as either uniformly distributed in the cytoplasm or in a retained form of polysaccharide-lignin lattice via hydrogen bonds. Therefore, such analytes are not able to be extracted by conventional methods. EAE is treated as a potent mechanism which favours the seperation of the firm linkages between the cell wall, BA and some other nutrients (Rosenthal et al., 1996). EAE has been implemented as enzyme assisted aqueous extraction (EAAE) and enzyme assisted cold pressing process (EACP) (Latif and Anwar, 2009). The extraction of oil from plant seeds may be an example for the application of EAAE (Hanmoungjai et al., 2001; Rosenthal et al., 1996, Sharma et al., 2002). In contrast, EACP is implied unless polysaccharide-protein colloids are formed (Concha et al., 2004). In EAE, the process variables that must be controlled are reported as the enzyme concentration and composition, the size of plant particle, S/L ratio, duration of hydrolysis (Niranjan and Hanmoungjai, 2004). In addition, the moisture content of plant material was notified as a significant factor for the extraction (Dominguez et al., 1995). Besides, using water instead of organic solvents in EAE is an evidence for environmental concern (Puri et al., 2012).

Supercritical fluid extraction: In nature, the matter is present in solid, liquid and gaseous state. However, when the substances are held at a temperature and pressure above the critical point, a new phase called supercritical phase will be observed. In this phase, the particular properties of gas or liquid disappear which means that the supercritical fluid can not be liquified anymore by the change in pressure and temperature. Supercritical fluids both exhibit some features specific to gases such as diffusivity, surface tension and viscosity and also some characteristics of liquids such as solvation power, density. Thanks to these features, extraction of BA can be performed effectively (Sihvonen et al., 1999). Low viscosity and high diffusivity aid the supercritical fluid to diffuse into solid material easily and to attain the target concentrations during the reactions. Moreover, using supercritical fluids instead of organic solvents provides more rapid extraction (Artık et al., 2016). Simultaneously, the less energy requirement of SFE can make it more desirable in contrast to other extraction methods. Nevertheless, the solubility of every compound does differ in the type of supercritical fluid. Hence, the proper selection of solvent is a crucial step for SFE. Mostly, the

supercritical fluids with non-explosive, environmentally safe, inexpensive properties are chosen as solvent. Hence, carbondioxide is the most preferable solvent among other supercritical fluids like ammonia, n-butane, methane, ethane, nitrousoxide, chlorotrifluoromethane, methanol, acetone and ethanol. The supercritical carbondioxide is a primarily selective solvent for hydrocarbons with a molecular weight lower than 250 and those oxides of monoterpenes and sesquiterpenes with a range of molecular mass between 250 and 400. On the other hand, the solubility of polyphenols with a molecular mass greater than 400 in supercritical CO_2 is poor (Artık et al., 2016). For this reason, the solubility of some compounds in supercritical CO_2 can be enhanced using several modifiers, in other words co-solvents (Lang and Wai, 2001; Ghafoor et al., 2010). Methanol, ethanol, dichloromethane, ethylacetate are typical co-solvents used in SFE. Methanol with a co-solvent ratio up to 20% (the ratio of the co-solvent volume to the volume of solvent mixture) is suggested as one of the most effective modifiers. However, owing to its toxicity, ethanol may be selected as a modifier instead (Chiu et al. 2002; De Lucas et al. 2007; Sanal et al. 2005; Hamburger et al., 2004; Lang and Wai, 2001). The mass transfer rate and extraction efficiency of BA in the food matrix significantly relates to the co-solvent ratio (Artık et al., 2016). The solubility of catechins might be achieved with a modifier (ethanol) at a co-solvent ratio of 5-10% , while proanthocyanidins can not be extracted even by the use of the same modifier with a ratio of 15% (Murga et al., 2002).

In SFE, the generation of the supercritical fluid is performed by an equipment which can control pressure, temperature and flow rates of supercritical fluid and modifier (Artık et al., 2016). The parts of SFE equipment is shown in Fig 1.

Several process parameters which might be controlled in SFE during the extraction of BA are noticeable (Reverchon and Marco, 2006). These variables presumed to influence the yield of SFE are reported as temperature, pressure, size of the particle, moisture content of the sample, extraction time, CO_2 mass flow rate and solute to solvent ratio (Temelli and Güçlü-Üstündag, 2005; Ibanez et al., 2012).

Accelerated solvent extraction: When the extraction is carried out with solvents at a temperature and pressure lower than their critical points, this method is defined as Accelerated Solvent Extraction (ASE) or Pressurized Liquid Extraction (PLE). This method provides an effective penetration of the solvent into plant tissue at elevated temperature and pressure under nitrogen atmosphere and thereby hinders the degradation of phenolic compounds.

The elevated pressure gives rise to the stable liquid form of the solvent even though the temperature exceeds its boiling point (Richter et al., 1996). It also ensures rapid contact of the solvent with food matrix by collapsing the bubbles in the sample (Richter et al., 1996; Dawidowicz et al., 2006; Pavlovic et al., 2007). However, it is considered to have a minor effect on the extraction yield (Carabias-Martinez et al., 2005). High temperature practises in ASE assist to obtain higher extraction yields by van der Waals forces, hydrogen bonding and dipole-dipole interactions. Rise in temperature leads to the reduction in the viscosity of the solvent and facilitates the diffusion. Besides temperature and pressure, other factors such as extraction time, selection of the solvent, flow rate of solvent, amount of sample, the position of the analyte within the food matrix, number of extraction cycles, extraction mode (dynamic or static), type and ratio of modifiers have impact on the performance of ASE (Sun et al., 2012; Çam and Hışıl, 2009). In addition, the sample composition, moisture content of the food matrix, size of the particles, pretreatment (drying, milling) conditions belong to the governing factors of ASE (Artık et al., 2016). Among all the critical parameters, type of solvent and temperature are reported as the primarily effective factors for ASE (Çam and Hışıl, 2009).

ASE equipment basically consists of an electrovalve, thermostatted extraction chamber, a pump, a pressurizing unit and a collector (Benthin et al., 1999). ASE has been reported a superior technique in comparison with the conventional solvent extraction due to shorter extraction time and less consumption of solvent (Richter et al., 1996). As reported by Lee and Kim (2010), bioactive lignans can be effectively extracted from plant tissue at 125°C in a 5 minute static time which is substantially shorter compared to SOE and UAE (3 hours). In the extraction of carotenoids, ASE was reported as a more efficient extraction system compared to traditional extraction methods since less volumes of solvent were required for a shorter extraction period (Denery et al., 2004). Besides, it is proposed as an alternative to SFE due to its capability of extracting polar compounds (Kaufmann and Christen, 2002). As SFE is a satisfactory solution for the extraction of BA with non-polar characteristics and the use of a co-solvent is necessary for the extraction of polar compounds, there can be no use of any other solvent in ASE.

In addition, enhanced mass transfer and solubility can be achieved at higher temperatures than the atmospheric boiling point of the solvent. When the temperature is increased from 100 to 250°C at 20 MPa, it was observed that the dielectric constant of water decreased significantly which is evident for the decrease in polarity of the solvent above its boiling point than the polarity of solvent at room temperature (Kim et al., 2009). Thus, one might expect that the lower the polarity of the solvent, the higher the solubility.The high solubility phenomenon can be associated with the low dielectric constant and low polarity of the solvent since higher extraction yields were obtained while working with polycyclic aromatic hydrocarbons, such as chrysene, propazine, and chlorothalonil (Miller et al., 1998). When distilled water is used as solvent in ASE, the polarity of water falls within the range of 100-374°C and at high pressure. Thanks to this features, favourable conditions might be provided which allow the extraction of compounds with high, medium and low polarity (Mustafa and Turner, 2011). This method is called subcritical water extraction (SWE). The governing factors in SWE can be summarized as mass of sample, sample composition, solvent volume and flow rate, temperature, pressure and duration of the process.

High hydrostatic pressure extraction: High hydrostatic pressure extraction (HHP) is treated as one of the most recent novel techniques which includes the non-thermal application at ultra high pressures (1000-8000 bar) on mass transfer basis. The system operates at room temperature. When pressure is exerted, according to the mass transfer phenomena and phase theories, the permeability of plant tissue rises and the diffusivity of cell components is facilitated (Zhang et al., 2005). The enormous pressure gradient between inside and outside the cell helps the solvent to diffuse into the cell which results in the motion of cellular components out of the cell (Zhang et al., 2005). HHP method promotes the extraction yield, decreases the selectivity of the cell and thus leads to cell deformation and protein denaturation (Jun et al., 2009).

The recent studies revealing the conditions and results of novel extraction methods on BA are summarized in Table 3.

Overall Evaluation of Novel Extraction Methods

When it comes to an overall evaluation, one should obviously notify that novel extraction methods tend to reduce the operational costs by saving time and solvent. SLE, HD and SOE can take hours even a day to extract the analyte from the food matrix completely. In addition, acidic or alkaline treatment may be required in some cases which means a more severe environmental pollution and waste disposal. On the other hand, the specificity of the analyte, the target of the study (whether it aims to determine maximum extraction yield, a great deal of diversity of BA, minimum damage in the food matrix and its sensory properties or not) and the feasibility of the technique should be in accordance with each other. That is, essential oils can be extracted using SOE, and/or HD, SFE. ASE can be selected for the extraction of flavonoids with respect to the extraction yields.

Total costs of the extraction can be grouped into two categories as investment costs and operational costs. Moreover, the yield of extraction per unit mass of sample does significantly influence the total costs. Compared to HHP, PEF, ASE and SFE, UAE is more economical since the equipment costs are relatively lower. The aid of mechanical stirring provides a more uniform structure of the extract in UAE and thus increases the extraction efficiency. However, energy and solvent consumption, a post-filtration requirement in UAE might increase significantly its processing costs (Chemat et al., 2011). Furthermore, it is obvious that the higher the pressure and the temperature, the greater the expenditures for energy and purged gases consumed during ASE, SFE and HHP. Besides the application of elevated temperatures might lead to a substantial loss of heat sensitive compounds and consequently lower extraction yields which inçline unit costs per sample.

One should notice that on the basis of a comparison with the extraction techniques requiring high investment costs, PEF is more likely to be a better choice due to cleaner and safer extraction technology compared to ASE, SFE, HHP since the damage in the food matrix is minimized and the changes in the sensory, physical chemical characteristics of the food are significantly prevented in addition to the avoidance of solvent consumption. Even though PEF systems require high level of capital costs, the electricity costs are diminishingly increased by larger capacities which is directly proportional to the square root of the capacity ($/liter/hour). In PEF treatment, a 10× higher capacity system will be approximately 3–3.5× the capital cost. This relationship holds for the range of pilot and commercial systems possible (~20 kW–1 MW) (Kempkes and Tokusoglu, 2014). This might be an evidence that industrial PEF systems can be more economical than pilot scale equipments and therefore more preferable on industrial scale compared to those of other novel extraction methods.

From the eco-friendliness point of view, SFE might be considered as an alternative to PEF since the extraction is carried out with non-toxic and inflammable solvents which are eliminated easily after the extraction. Lower sample size, lower amount of solvent consumption and high level of selectivity are the other benefits of SFE compared to the other novel extraction methods such as UAE, MAE and ASE (Chemat et al., 2011).

In traditional extraction systems and in some applications of MAE (SMAE and standard type of MAE), it seems that the food sample might be more frequently exposed to air and light since they are designed as open atmosphere systems. Hence, the oxidation of target compounds and the interactions of non-desirable oxidative compounds with the analyte might occur which also reduces the accuracy of the analysis and the extraction yield (Chan et al., 2011).

In contrast to MAE, more rapid (Lopez-Avila et al., 1996) and simpler extraction (Luque de Garcia and Luque de Castro, 2003) is carried out by using UAE without any considerations in relation to the restrictions about the polarity of solvent, type of matrix and the moisture content of the food sample (Chemat et al., 2011).

MAE and UAE are the extraction methods which offer multiple choices of operation modes for a wide variety of BA and food types so that the user can easily select and adapt the method in combination with each other and other extraction methods. Thus, the possible drawbacks of other techniques might be eliminated thanks to this feature. Nevertheless, the cost effectiveness of the combined extraction systems must be taken into account before these are utilized on industrial scale since the modifications strongly depend on the additional costs of installation and maintenance which is in relation with the marginal costs of the final product. Furthermore, the restrictions of MAE and UAE such as oxidation risk and thermal degradation must be taken into consideration so that the combined systems can be feasible.

When one wants to focus on the extraction techniques based on industrial scale, several attempts related to the extraction of BA are to be recognized. Decaffeination of coffee (Mawwell, USA) can be given as an example (IFS, 2006). The low temperature application by using SFE provides a full flavor aromatic profile of coffee without caffeine. In Germany, there is a plant able to decaffeinate approximately 27.3 million kg of product per year (Mc Nally, 2000). Nearby, the extracted caffeine can be used for different purposes in the food market (IFS, 2006). Besides Diam Bouchage (France) produces a special cork flavor made of Trichloroanisole extracted by SFE (IFS, 2006).

Table 3 Previous research based on the novel extraction techniques for BA from plant materials

Method	Analyte	Plant	Conditions	Result	Reference
ASE, UAE	Chlorogenic acid	Eggplant	80% metanol; 50% metanol; Acetone ASE: 70 bar; 100°C, time: 30 min., static mode: 5 min; Cycles: 4 UAE: for 15 min. in sonicator bath at ambient temperature	ASE: 22 mg/L; UAE: 18 mg/L chlorogenic acid. Extraction yield and recovery of chlorogenic acid by ASE is the highest compared to UAE and other conventional methods	Luthria and Mukhopadhyay (2006)
SFE	Anthocyanin	Potato peel	100, 400 bar at 35 and 65°C with ethanol (5% v/v) as co-solvent	Max. anthocyanin yield was achieved at 100 bar and 65°C.	Cardoso et al. (2013)
HHP, UAE	Phenolics	Green tea leaves	HHP at 5000 bar with Ethanol (50% v/v) S/L: 1/20 UAE: 250 W, 50 Hz for 90 min. at 20-40^{0}C with Ethanol (50% v/v)	Extraction yield of polyphenols by HHP and UAE 30% and 29% respectively	Jun et al. (2009)
MAE, UAE	Phycocyanin	Cyanobacteria	MAE: 50-150-250 W 1.5% $CaCl_2$ (w/v) At 10-30-60-180-300-600 s of time intervals UAE: 90-150-230 W At 5-10-15-20-25-30 min. of time intervals	Max. phycocyanin: MAE: 150 W-600 s.-1.5% $CaCl_2$ (w/v): 110.2 mg/g on d.b; UAE: 25 min. 1.5% $CaCl_2$ (w/v)- 100 mg/g on d.b.	Ilter and Ertekin (2017)
SFE	Essential oils	*Lavandula viridis*	At 40°C, 12 and 18 MPa with two seperators, CO2 flow rate: 0.3 kg/h	Extraction yield: 9.27% and 8.80% from second seperator at 12 and 18 MPa respectively	Costa et al. (2012)
HHP	Lycopene	Tomato paste waste	Ethanol 45%-95%v/v), chloroform, water at 100-600 MPa for 1-10 min. S/L: 1/1 to 1/8 g/ml	The highest recovery (92%) was at 500 MPa pressure,1 min duration, 75% ethanol concentration, and 1:6 (g/ml) solid/liquid ratio	Jun (2006)
PEF	Phenolics, Anthocyanin	Red cabbage	1 kV/cm, 20 pulses for 30 ms	2.5 and 1.85 times more phenolics and anthocyanins than yielded by SLE respectively	Kannan (2011)
ASE, UAE	Isoflavones	Soybean	ASE: 1000 psi at 100°C, with a 5 min equilibration time, a 7 min static time, a 90 s purge time with three extraction cycles UAE: Sonicator bath at ambient temperature for 15 min.	Optimum total isoflavones recoveries from soybean samples were yielded with dimethyl sulphoxide: ethanol: water (5:75:25, v/v/v) solvent mixture using ASE.	Luthria et al. (2007)
SFE	β-Carotene	Carrot	At 313-343 K; 27.6–55.1 MPa Co-solvent: 5% canola oil	171.7–899.97 µg/g feed	Sun and Temelli (2006)
EAE	Ferulic acid, vanillic acid, vanillin, cinnamic acid	Sweet potato	Ultraflo L; Viscozyme L; α-Amylase at conc. of 0- 0.1-0.5-1% 1-12 h 37°C	The rate of release of ferulic acid is optimal, when Ultraflo-L (1%) was used. For the release of vanilic acid Viscozyme-L is most effective.	Min et al. (2006)
PEF, HHP, UAE	Phenolics, anthocyanin	Grape skin	HHP:50°C 600 MPa, Etanol PEF: 3 kV/cm -30 pulses UAE: 35 kHz, 70°C for 1 h in ultrasonic bath	Total phenolics: PEF (4 times) > HHP (3 times) > UAE (2 times) as compared to SLE. Anthocyanin recovery by PEF is 10% higher than HHP and 17% higher than SLE	Corrales et al. (2008)
SWE	Anthocyanin, phenolic acids, flavonols	Red grape skin	at 100, 110, 120, 130, 140, 150 and 160°C for 40 s. With methanol (60% v/v) and subcritical sulphured water extraction (SSW)	Extraction temperatures greater than 110°C resulted in decreased contents of individual and total anthocyanins.	Ju and Howard (2005)
HHP and SFE	Phenolics	Sour Cherry, peach, apple pomace	HHP: 50-125-200 MPa; 20-40-60°C; 10-25-40 min. SFE: 20-40-60 MPa, 40-50-60°C 10-25-40 min.	Optimization for sour cherry pomace: SFE-20% etanol-40 min. at 55-59 MPa and 50-54.4°C Total phenolics: 0.6 mg GAE/g on d.b. HHP: 176-193 MPa, 60°C, 25 dk. Tot. Phenolic: 3.8 mg GAE/g on d.b.	Adil and Bayındırlı (2006)

The first commercial applications based on PEF were reported by Kempkes and Tokuşoğlu (2014). The extraction of lipids from algae for production of biofuels was successfully carried out by the installation of PEF equipment. Kempkes (2017) reported that more than one half of approximately 100 PEF systems operating in the world in 2015 have been utilized for industrial purposes. Most of these commercially designed equipment have been made use of for tissue modifications such as extraction, drying, tissue softening and juice production with extended shelf life. However, it is noteworthy that no commercial PEF extraction system for the use in food processing has been reported so far even though the progressive research on this field reveal that several instruments which are more likely to be compared with PEF systems in potato chips processing from processing costs and energy efficiency points of view might be improved and adopted in near future (Kempkes, 2017).

UAE has also been reported as one of the extraction methods feasible for industrial applications. REUS a company in France developed reactors by a volume range between 30 and 1000 liters with double mantles that does not allow the temperature inside the sample to rise (Chemat et al., 2011). Giotti and Moliserb are the other examples for UAE industrial applications that perform extraction at nearly room temperatures so that the antioxidants and essential oils can be extracted without being damaged (Chemat et al., 2011). A number of different ultrasound reactors available in the food industry have been reported by Vinatoru (2001) and Chisti (2003). These include a) stirred ultrasound horn directly submerged into stirred bath, b) stirred reactor with ultrasound coupled to the vessels walls and (c) recycling of product from stirred reactor through an external ultrasonic flow-cell. These modifications allow intense and permanent ultrasonic power with a wide range of applications from low intensity in a large volume reactor (0.01 to 0.1 W/cm^3) to high intensity (1 to 10 W/cm^3) in an external flow-cell (Vilkhu et al., 2008). Furthermore, energy efficiency and economic considerations have been regarded as the key aspects for the selection of these mixed frequency ultrasonic reactors. High energy efficiency (85% of the power is transferred into the medium), moderate investment costs including development, capital and installation costs (700000 $ per year), short payback periods (4 months) are the remarkable economical factors that could make the choice of an extraction system in favour of UAE on industrial scale (Patist and Bates, 2008).

Mustafa and Turner (2011) reported that no commercial applications of ASE running at dynamic mode are available. Though, an instrument designed by Dionex Corporation (ASE-350) provides persistent fresh solvent flow which could be promising for future aspects (Mustafa and Turner, 2011) .

The industrial application of HHP has been performed in Japan, United States and Europe basically for pasteurization of food products. The technique was first introduced in Japan by the leading firms of the market called Mitsubishi Heavy Industries Ltd, Kobe Steel Ltd., Nippon Steel Ltd. Three major types of industrial HHP systems are present: Batch, continuous and semi-continuous. Batch type of equipment is convenient for solid and liquid products. However, continuous and semi-continuous processes are applicable for only liquid foods (Elamin et al., 2015). The cost of commercial-scale HHP equipment might range from 500,000 to over 2.5 million US Dollars, depending on the equipment capacity and the level of automation used (Koutchma, 2014; Sampedro et al., 2014). Owing to the high capital costs made up of 80% of total costs and processing costs including labor, utilities, maintenance (Elamin et al., 2015), the feasibility of HHP on industrial scale seems to be restricted for the near future.

Conclusion

Despite the traditional extraction methods have still been widely used, the following approaches in relation to the advanced techniques come into prominence:

- Using samples in low quantities
- Higher selectivity of the method
- Conformance with the automation
- Minimizing solvent consumption and wastes leading to environmental problems (Smith, 2003).

Consequently, the novel methods reduce the extraction time and solvent consumption. PEF and HHP are recognized as effective and environmentally friendly techniques inspite of high investment costs. The most convenient methods for the extraction of phenolic acids seem to be HHP, ASE and SFE methods. For anthocyanins and tannins, satisfactory results can be achieved by SLE and SWE techniques. From the perspective of efficiency in extraction of EO, HD and SFE methods are realizable. MAE and UAE promise the extraction of a diverse range of BA from a wide variety of food matrices at moderate operational costs with multi-optional applications.

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