GIDA
THE JOURNAL OF FOOD
E-ISSN 1309-6273, ISSN 1300-3070

Review / Derleme
GIDA (2025) 50 (1) 56-73
doi: 10.15237/gida.GD24094

A REVIEW OF ULTRASOUND-ASSISTED EXTRACTION OF BIOACTIVE COMPOUNDS FROM COFFEE WASTE

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Received/Geliş: 09.09.2024; *Accepted*/Kabul: 07.01.2025; *Published online*/Online baskı: 28.01.2025

Özdemir, M., Yıldırım, R., Yurttaş, R., Başargan, D., Hakcı, M. B. (2025). A review of ultrasound-assisted extraction of bioactive compounds from coffee waste. GIDA (2025) 50 (1) 56-73 doi: 10.15237/ gida.GD24094

Özdemir, M., Yıldırım, R., Yurttaş, R., Başargan, D., Hakcı, M. B. (2025). Kahve atıklarından biyoaktif bileşiklerin ultrases destekli ekstraksiyonu. GIDA (2025) 50 (1) 56-73 doi: 10.15237/ gida.GD24094

ABSTRACT

The objective of this paper is to review the effectiveness of ultrasound-assisted extraction, particularly targeting phenolic and flavonoid compounds from coffee waste. The mechanism, advantages, disadvantages and some of the important factors affecting ultrasound-assisted extraction are discussed. Previous studies and current applications of ultrasound-assisted extraction on the extraction of phenolics and flavonoids from various coffee wastes are reviewed. Ultrasound-assisted extraction is easier to use, can be done at the room temperature, increases efficiency, utilizes less solvent and energy, reduces operating costs, and better preserves of the bioactivity of thermosensitive compounds. This review shows that key parameters affecting the extraction of bioactive compounds using ultrasound technology are temperature, contact time, type of solvent, solid to solvent ratio, ultrasonic power and ultrasonic frequency. In conclusion, all the reported applications reveal that ultrasound-assisted extraction stands out as an emerging and green extraction technique to extract phenolic and flavonoid compounds from coffee waste.

Keywords: Coffee waste, ultrasound-assisted extraction, phenolics, flavonoids

KAHVE ATIKLARINDAN BİYOAKTİF BİLEŞİKLERİN ULTRASES DESTEKLİ EKSTRAKSİYONUNUN İNCELENMESİ

ÖZ

Bu derleme makalesinin amacı, özellikle kahve atıklarından fenolik ve flavonoit bileşikleri hedef alan ultrases destekli ekstraksiyonun etkinliğini araştırmaktır. Ultrases destekli ekstraksiyonun mekanizması, avantajları, dezavantajları ve etkileyen bazı önemli faktörler tartışılmaktadır. Çeşitli kahve atıklarından fenolik ve flavonoitlerin ekstraksiyonu üzerine ultrases destekli ekstraksiyon ile ilgili çalışmalar ve mevcut uygulamalar verilmektedir. Ultrases destekli ekstraksiyonun kullanımı daha kolaydır, oda sıcaklığında yapılabilir, verimliliği artırır, daha az çözücü ve enerji kullanır, işletme maliyetlerini azaltır ve ısıya duyarlı bileşiklerin biyoaktivitesini daha iyi korur. Bu derleme çalışması,

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ultrases teknolojisi kullanılarak biyoaktif bileşiklerin ekstraksiyonunu etkileyen temel parametrelerin sıcaklık, temas süresi, çözücü türü, katı-çözücü oranı, ultrasonik güç ve ultrasonik frekans olduğunu göstermektedir. Sonuç olarak, mevcut tüm uygulamalar, ultrases destekli ekstraksiyonun kahve atıklarından fenolik ve flavonoit bileşiklerin ekstraksiyonu için yeni ve yeşil bir ekstraksiyon tekniği olarak öne çıktığını ortaya koymaktadır.

Anahtar kelimeler: Kahve atığı, ultrases destekli ekstraksiyon, fenolikler, flavonoitler

INTRODUCTION

Coffee, discovered in what was then known as Abyssinia, present-day Ethiopia, around 700-800 AD, belongs to the *Rubiaceae* family under the Coffee genus (Worku, 2023). Gaining immense popularity worldwide, coffee is distinguished by two main types: *Coffea arabica* L. and *Coffea canephora*, known as Arabica and Robusta, respectively (Mensah et al., 2024). Arabica, constituting 75% of global coffee production, is favored for its unique flavor profile while Robusta, accounting for 23-24% of global coffee production, has a stronger taste, and holds significant importance in international coffee trade (Arya et al., 2022). Coffee, beyond being a beverage, has played a crucial role throughout history as a cultural symbol, a meeting point for communities and an economic powerhouse. The coffee culture, originating in Ethiopia and spreading globally, has evolved with diverse variations in different regions and cultures, becoming an indispensable beverage contributing to the global economy (Alves et al., 2017). Today, coffee continues to be one of the most consumed products in the world, and the popularity of coffee is predicted to increase more in the future (McNutt and He, 2019).

An often-overlooked fact is the vast and often unused waste produced by the coffee industry (McNutt and He, 2019). These wastes include large amounts of by-products, including waste from post-harvest processing, coffee roasting and coffee consumption such as immature/defective beans, coffee husk, pulp, mucilage, parchment, silverskin and spent coffee grounds (Alves et al., 2017; Hoseini et al., 2021). Due to the high production rate in the coffee industry, the amount of these wastes is also high (Janissen and Huynh, 2018). The most abundant by-product of coffee is spent coffee grounds, produced by consumers in homes and cafeterias as well as during industrial production of the instant coffee industry (Martinez-Saez et al., 2017; Bondam et al., 2022).

Since coffee and coffee waste are rich in bioactive compounds, they have high antioxidant activity, and these compounds are extracted and used in the food and pharmaceutical industries (Panusa et al., 2013; Serna-Jiménez et al., 2022). Utilization of coffee waste will both enable the recovery of high value-added compounds from these wastes, reduce the environmental impact of coffee waste and contribute to the global economy (Johnson et al., 2022). For this reason, studies and practices aimed at reusing coffee waste are of great importance. Phenolic and flavonoid compounds have various physiological activities such as antioxidant, antimicrobial, antimutagenic, anti-inflammatory and antiallergenic and are used in food, nutraceutical, cosmetic and pharmaceutical industries (Nurzyńska-Wierdak, 2023).

Bioactive compounds from coffee waste including polyphenols and flavonoids are usually extracted using conventional extraction methods, the most common of which is solid-liquid extraction (Bondam et al., 2022; Lee et al., 2023). Traditionally, solid-liquid extraction is usually performed at high temperatures and pressures to extract phenolics and flavonoids from various coffee wastes. The use of high temperature with and without high pressure degrades heat-sensitive compounds such as phenolics and flavonoids (Bondam et al., 2022). Solid-liquid extraction is also characterized by low extraction yield, long extraction time, low selectivity and excess solvent consumption (Bouhzam et al., 2023; Hu et al., 2023). Supercritical fluid extraction, microwave-assisted extraction, pulsed electric field and ultrasound-assisted extraction are commonly used emerging and eco-friendly techniques for the recovery of bioactive compounds (Buvaneshwaran et al., 2023). Ultrasound-assisted extraction steps ahead among the aforementioned extraction techniques (Lee et al., 2023). It is simple, fast, low-cost and effective technique with high reproducibility rate, high extraction efficiency in a short time, lower energy output and applicability to diversified samples (Yang et al., 2024). The mechanism of this method depends on the effect of ultrasonic energy generated with frequencies higher than 20 kHz, where high-intensity sound waves induce the formation of microbubbles. These microbubbles create cavitation in the liquid medium, providing a high shear force, and when the bubbles burst, they cause micro-mixing and macro turbulence, providing a higher level of contact between solid and liquid (Hassan and Al-Yaqoobi, 2023). This causes a variety of effects such as erosion of the solid in contact, particle fragmentation, capillary action, surface peeling, tissue disruption and ultrasound perforation, thus all accelerating cell destruction and mass transfer (Chemat et al., 2017; Zahari et al., 2020).

Extracting valuable bioactive compounds from coffee waste is critical to improve the use of bioactive compounds in other foods, increase the valorization of coffee waste, develop more sustainable food processes, and implement new alternatives to manage coffee wastes and by-products derived from coffee processing. Therefore, the main objective of this study is to provide information on the mechanism, advantages and disadvantages of the ultrasound-assisted extraction, important factors affecting the ultrasound-assisted extraction of bioactive compounds and current applications of ultrasound-assisted extraction in extraction of phenolics and flavonoids from various coffee wastes. This review is believed to increase awareness of the recovery of phenolic and flavonoid compounds from coffee waste, ensure the valorization of various coffee wastes, and contribute to sustainability.

BY-PRODUCTS OF COFFEE PRODUCTION

Coffee industry mainly produces three types of coffee product: instant coffee, coffee drink and coffee bean while harvesting, post-harvest and by-products resulting from the roasting process and coffee consumption generate coffee waste (Figure 1). The coffee harvesting and roasting processes yield immature and defective beans, respectively. These low-grade coffee beans must be separated from the mature and non-defective beans because they reduce the quality of the final products. The defective green coffee beans had the total phenolic content (TPC) of 65.4 mg chlorogenic acid equivalent (CGAE)/g of dry weight (Machado et al., 2023), which was approximately 30% lower than the TPC of non-defective green coffee beans (94.2 mg CGAE/g of dry weight) (Pimpley and Murthy, 2021). The total flavonoid content (TFC) of defective green coffee beans was reported to be 52.3 mg catechin equivalent (CE)/g of dry weight (Machado et al., 2023), which was identical to the TFC of non-defective green coffee beans (52.1 mg CE/g of dry weight) (Abdeltaif et al., 2018).

The pulp, mucilage and parchment surround the coffee bean, and they are rich in various components such as proteins, carbohydrates, fats, minerals and caffeine (Alves et al., 2017; Oliveira et al., 2021). The coffee pulp is covered by an outer skin that resembles that of a cherry. The outer skin is normally removed from the coffee fruit after it has been picked. Coffee pulp is a by-product of the wet processing method in coffee hulling process (Oliveira et al., 2021). Coffee pulp has a high moisture content due to the inclusion of water during the washing of coffee cherries before pulping, and mainly rich in carbohydrates, proteins and minerals, and contains significant amounts of tannins, polyphenols, dietary fiber and caffeine (Murthy and Naidu, 2012; Kovalcik et al., 2018). Geremu et al. (2016) determined that the TPC recovered from different coffee pulp extracts varied from 489.5 to 1809.9 mg gallic acid equivalent (GAE)/g of wet weight depending on the source of the pulp, solvent type and solvent concentration. Similarly, the TFC values of the coffee pulp ranged from 0.79 to 25.1 mg CE/g of dry weight, with the lowest TFC using ethyl acetate and the highest TFC using ethanol as the extraction solvent, respectively (Chen et al., 2021). The fruity layer of the coffee cherry that lies between the outer skin and the parchment layer enclosing the coffee bean is known as mucilage.

Mucilage contains both simple and complex sugars that can be used in fermentation. Mucilage is also rich in polyphenols and flavonoids where it has the TPC of 1618 mg GAE/g of dry weight and TFC of 532 mg quercetin equivalent (QE)/g of dry weight (Kc et al., 2021). Fibrous endocarp, known as parchment, covers both hemispheres of the coffee bean and separates them from each other (Iriondo-DeHond et al., 2019). It is rich in cellulose, hemicellulose and lignin (Klingel et al., 2020). It was reported that the TPC of the coffee parchment ranged from 1.2 to 3.1 mg GAE/g of dry weight (Benitez et al., 2019). Aguilera et al. (2019) determined that the TPC and TFC of coffee parchment varied from 0.72 to 2.04 mg GAE/g of dry weight and 0.15 to 1.61 mg CE/g of dry weight, respectively. In wet coffee processing, the parchment is removed after drying and hulling processes (Janissen and Huynh, 2018).

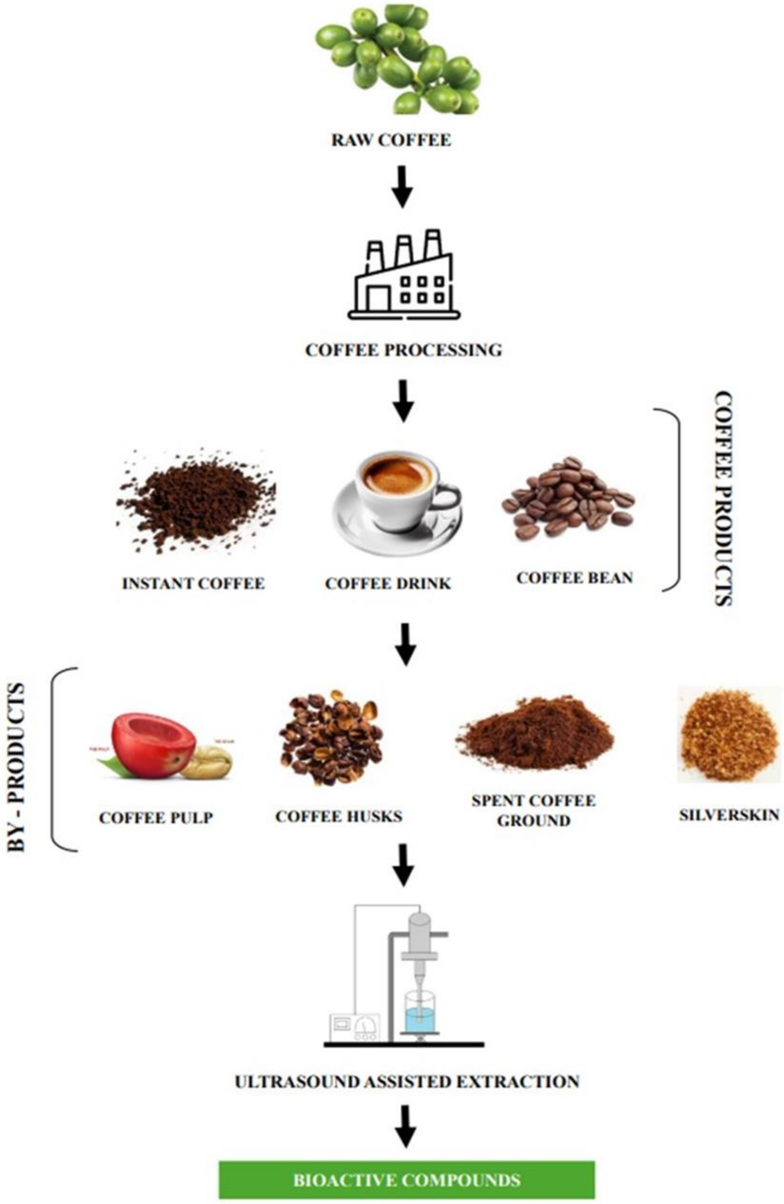

Figure 1. Coffee products and by-products

Coffee husk is a by-product of the dry processing method in coffee hulling process (Oliveira et al., 2021). The coffee husk contains pulp, pectin and parchment. Therefore, carbohydrates are higher than those of the coffee pulp (Hoseini et al., 2021). Approximately 0.18 tons of coffee husks are obtained from one ton of coffee fruit (Oliveira et al., 2021). The TPC of the coffee husk varied between 15.6 mg CGAE/g of dry weight (Iriondo-DeHond et al., 2019) and 97.9 mg CGAE/g of dry weight (Silva et al., 2020) while the TFC of the coffee husk changed from 0.2 to 15.7 mg CE/g of dry weight (Silva et al., 2020). The variations in the TPC and TFC values are due to various factors, including the measurement method, extraction method, extraction solvent, and geographical origin or species of the coffee beans.

Coffee silverskin, a by-product obtained during roasting of green coffee beans, is an extremely thin layer sticking to the coffee beans and is separated during coffee roasting. One ton of coffee silverskin is produced from every 120 tons of roasted coffee (Martuscelli et al., 2021) and considering that coffee consumption is constantly increasing, coffee silverskin can be considered to have great potential for the extraction of bioactive compounds. Coffee silverskin contains melanoidins and phenolic compounds resulting from Maillard reactions during roasting, so it exhibits antioxidant activity and it is used as a functional ingredient in the food industry (Narita and Inouye, 2014). Costa et al. (2014) extracted the total phenolic compounds from the coffee silverskin using different combinations of solvents, temperatures and extraction times, and reported a TPC of 16 mg GAE/g of dry weight. The TFC of the coffee silverskin changed from 1.9 to 8.6 g CE/g of dry weight for different varieties and roasting times where longer roasting times resulted in coffee silverskin with higher TFC (McDonald et al., 2022).

To obtain a high-end coffee experience, roasted coffee powder is contacted with hot water or steam to release various volatile and non-volatile compounds that give the flavor, aroma and taste to the coffee appreciated by the coffee drinkers worldwide (Zuorro and Lavecchia, 2013). Regardless of any extraction method used for coffee brewing, this process produces a solid waste called coffee ground (Severini et al., 2017). Spent coffee grounds are the most abundant by-product obtained as a result of coffee brewing at homes, cafeterias, restaurants, fast-food restaurants and during the extraction process for industrial instant coffee production (Kovalcik et al., 2018). These spent coffee grounds have a dark brown color, coarse texture and high moisture content (Alves et al., 2017). Coffee production generates approximately 6 million tons of coffee grounds each year (Franca and Oliveira, 2022; Yusufoğlu et al., 2024). Approximately 650 kg of coffee grounds are produced from one ton of green coffee, and approximately 2 kg of wet coffee grounds are generated per kilogram of soluble coffee produced (Lauberts et al., 2023). Coffee grounds are usually disposed of in landfills or incinerated, and this leads to undesirable consequences in terms of economic and environmental impacts (McNutt and He, 2019; Battista et al., 2020). However, spent coffee grounds can be mixed with other ingredients and especially used in cakes, cookies, muffins, biscuits, breads, yogurts, and fermented and distilled beverages (Martinez-Saez et al., 2017; Arya et al., 2022; Benincá et al., 2023; Dauber et al., 2024). Coffee ground cakes have the potential to be used as a source of fat-soluble vitamins (Hoseini et al., 2021).

There are many natural compounds in spent coffee grounds such as polyphenols, flavonoids, proteins, sugars, lignin, cellulose and hemicellulose (Loarca-Piña et al., 2015). Based on the extraction conditions and coffee variety, the TPC of spent coffee grounds varied from 9 to 29 mg GAE/g of dry weight (Zuorro, 2015; Choi and Koh, 2017; Ramón-Gonçalves et al., 2019).The phenolic composition of spent coffee grounds mainly includes chlorogenic acids (85%) such as 3-O-caffeoylquinic acid, 4-O-caffeoylquinic acid and 5-O-caffeoylquinic acid as well as caffeic acid (6%), and chlorogenic acid is the main substance with antioxidant activity in the spent coffee grounds (Okur et al., 2021). The most important material found in spent coffee grounds, aside from chlorogenic acid and its derivatives, is caffeine (Janissen and Huynh, 2018). The high concentrations of caffeine found in spent coffee grounds highlight the waste material's great potential as a natural source of phenolic antioxidants (Loarca-Piña et al., 2015). The caffeine in spent coffee ground is completely broken down by *Pleurotus ostreatus* LPB 09 fungal cultures, enabling spent coffee ground to be utilized as a cheap substrate for edible mushrooms and mushroom cultivation without the need for any pre-processing (Carrasco-Cabrera et al., 2019).

ULTRASOUND-ASSISTED EXTRACTION

One unique and highly promising extraction technique for obtaining phenolic and flavonoid compounds from coffee and coffee by-products is ultrasound-assisted extraction (Solomakou et al., 2022; Buvaneshwaran et al., 2023; Lee et al., 2023). Its user-friendly application and simple instrumental requirements make ultrasound-assisted extraction a desirable extraction method. This method effectively extracts chemicals from a solid matrix by using high-frequency sound waves and a small amount of solvent. There are two main types of ultrasounds used in industry: high-intensity low-frequency ultrasound (20 kHz $< f \leq$ 100 kHz) and low-intensity high-frequency ultrasound ($f >$ 100 kHz) (Chávez-Martínez et al., 2020). Low-intensity ultrasound is a technique in which ultrasonic wave propagates without changing the physical or chemical properties of the material. On the other hand, high-intensity ultrasonic waves create a distortion effect within the matrix by forming intense pressure and temperature gradients caused by cavitation bubbles. These bubbles coalesce and grow, and then collapse during the compression phase. As a result, progressive expansions and compressions accelerate or increase the rate of biochemical reactions and facilitate the release of bioactive compounds from plant tissues (Chemat et al., 2017).

Ultrasound-assisted extraction is generally performed using an ultrasonic bath (Figure 2) or ultrasonic probe (Figure 3). The solid matrix is distributed in a solvent in a stainless-steel tank that is attached to the transducer in the ultrasonic bath method. This method is economical and easy to use, but reproducibility can be low, causing limitations in the extraction process. Another technique is called ultrasonic probing, which involves attaching a transducer to a probe or horn. With little energy loss, the probe produces ultrasonic waves in the medium while submerged in the extraction vessel. Probe-based system is a good tool for the efficient extraction of bioactive compounds because they often have greater ultrasonic intensities. However, in this system, uneven energy distribution and decreasing power over time may reduce efficiency as compared to the bath system. In the probe system, the transmitted energy is focused on a specific sample region to create a more effective cavitation effect (Chemat et al., 2017).

Figure 2. Ultrasonic bath

Advantages and Disadvantages of Ultrasound-Assisted Extraction

Ultrasound-assisted extraction is a promising method to obtain bioactive compounds from various sources. Many advantages (Figure 4) offered by this method have led to its increasing use in industrial and scientific contexts. One of the main advantages of ultrasound-assisted extraction is its ability to greatly increase extraction efficiency. When ultrasonic waves are applied, mass transfer between the solvent and the sample matrix increases, leading to a more effective extraction than conventional extraction techniques. Additionally, ultrasound-assisted extraction uses less energy and materials, and shorter extraction time contributes to the economic sustainability of the process with improved efficiency. Ultrasound-assisted extraction is safe, inexpensive, reproducible and easy to use. It can be performed at room temperature and atmospheric pressure so heat-sensitive bioactive compounds can be extracted at low temperatures, which is very important in food and pharmaceutical industries to maintain the activity and integrity of heat-labile bioactive compounds. By dissolving cell walls and promoting the release of desired components, the ultrasound-assisted extraction can be tuned to extract specific molecules from complex matrices. On average, ultrasound-assisted extraction uses less solvent than conventional extraction methods, which reduces waste due to solvent generation and increases process sustainability, both of which are consistent with green extraction principles (Carreira-Casais et al., 2021). However, some disadvantages of ultrasound-assisted extraction should also be considered. Firstly, investment cost for the equipment can be high for large-scale industrial operations. Secondly, maintenance cost of the ultrasound equipment is another issue to consider because frequent maintenance is required to ensure that equipment is working properly and to prevent it from becoming unusable. Additionally, careful parameter tuning is required to balance extraction efficiency with compound stability due to the possibility of degradation of bioactive compounds caused by inappropriate or extreme ultrasonic conditions. Another factor to consider is cavitation, which increases mass transfer, but meanwhile it can cause physical damage or change in the chemical structure and bioactivity of the extracted substances. Additionally, in solid or dense matrices the penetration of ultrasonic waves may be limited, leading to insufficient extraction. Therefore, factors affecting the ultrasound-assisted extraction of bioactive compounds should be carefully considered, and responses should be optimized to obtain reliable and reproducible results.

Figure 3. Ultrasonic probe

Parameters Affecting Ultrasound-Assisted Extraction of Bioactive Compounds

Solvent type, solid-solvent ratio, extraction temperature, extraction time, ultrasonic power and ultrasonic frequency are important parameters influencing the ultrasound-assisted extraction of bioactive compounds (Figure 5) (Carreira-Casais et al., 2021; Ozdemir et al., 2024).

Solvent selection is crucial to maximize the solubility of the targeted bioactive compounds and optimize extraction efficiency. Many different solvents either alone or in combination with each other can be used in extraction of bioactive compounds where water or organic solvents like ethanol or methanol are frequently utilized. The biological material is usually chopped into small pieces or grinded for size reduction, thus increasing surface area (Shen et al., 2023). After the sample and solvent are mixed in a container, the system is subjected to ultrasonic waves, which allow the solvent to penetrate the material more effectively. It is crucial that the solvents used to extract the bioactive compounds from coffee grounds should be polar because polar solvents extract bioactive compounds much better than nonpolar solvents (Beaudor et al., 2023).

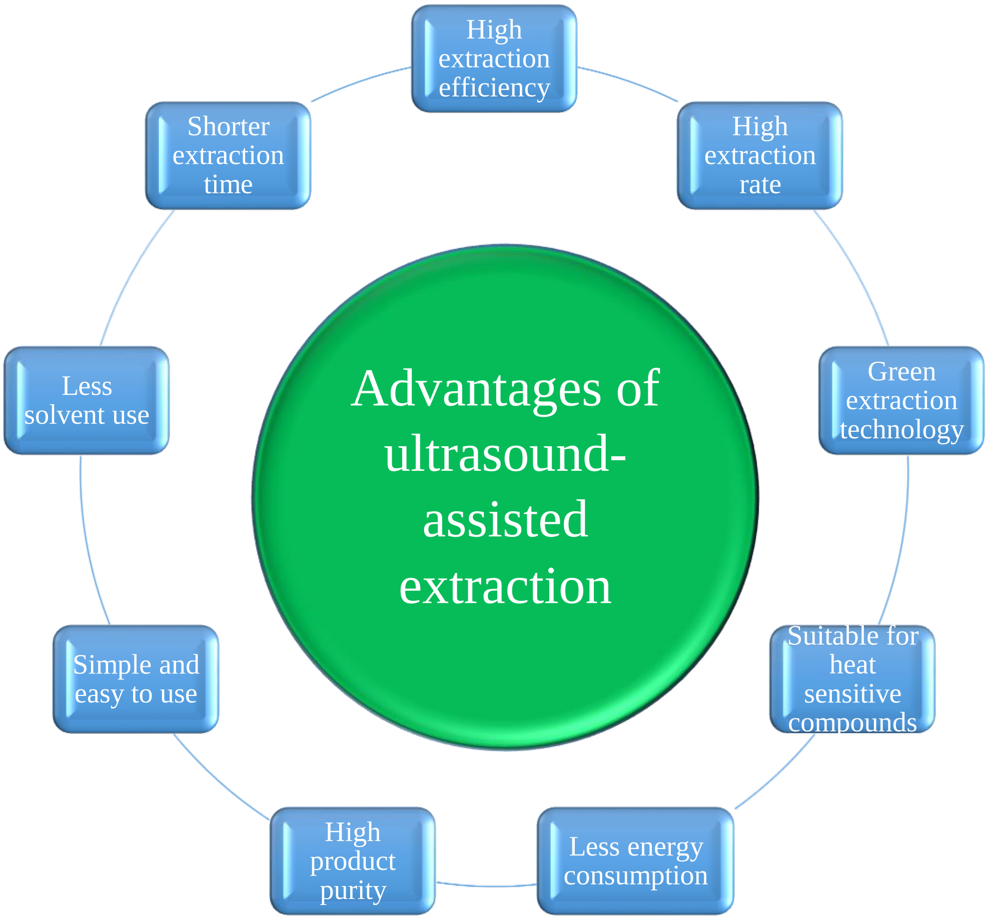

Figure 4. Advantages of ultrasound-assisted extraction

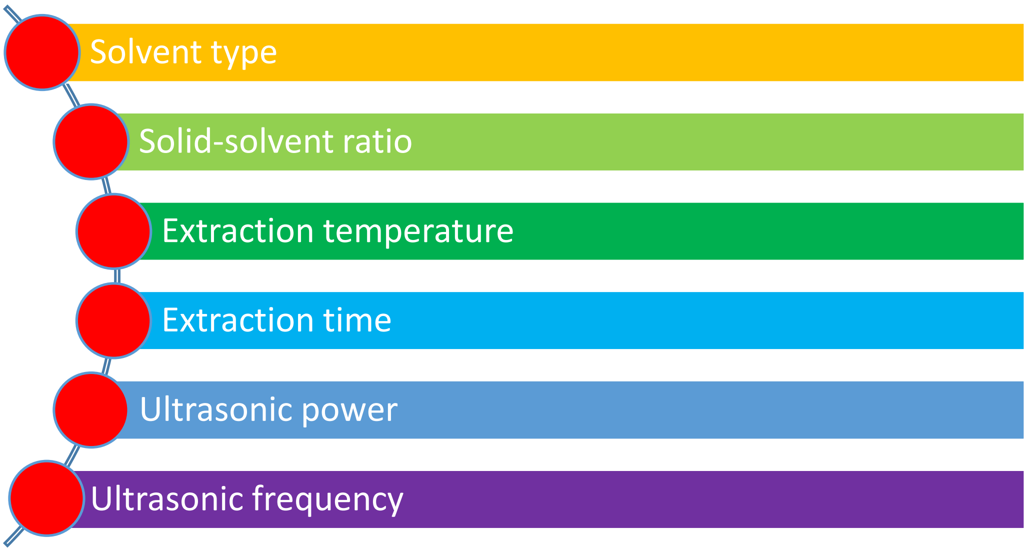

Figure 5. Factors affecting ultrasound-assisted extraction

One important parameter influencing the extraction efficiency is the ratio of solid to solvent. Although the optimized solid to solvent ratio guarantees the use of adequate solvent, using too much solvent might have negative effects on the environment and raise process costs. Therefore, optimizing the amount of solvent is important for the economic feasibility and environmental sustainability of the extraction process (Myo and Khat-Udomkiri, 2022). This ensures an overall improvement in the efficiency of the extraction process, enhances the amount of the extracted material, and improves the quality of the final product. On the other hand, higher solvent concentrations can generally result in higher extraction yields and efficiencies, but excessive concentrations can cause solubility issues and an increase in the viscosity of the solution (Al-Dhabi et al., 2017). Additionally, bioactive compounds may degrade if the solvent concentration is excessive (Beaudor et al., 2023).

Raising the temperature can improve mass transfer by making the target molecules more soluble in the solvent (Zamanipoor et al., 2020). As a result of the increased solubility, more bioactive compounds are dissolved from the wasted coffee matrix, thus improving extraction efficiency. Furthermore, high temperatures also increase the diffusion of bioactive substances into the solvent (Oroian et al., 2020). Higher temperatures lead to higher yields at shorter extraction times and faster extraction rates. Elevating the temperature facilitates the penetration of solvent into the wasted coffee matrix. Higher temperatures provide greater accessibility of the bioactive compounds by facilitating the interaction between the solvent and the target bioactive compounds, hence further enhancing extraction efficiency (Jha and Sit, 2022). On the other hand, excessive temperatures may cause heat-sensitive bioactive substances in the wasted coffee to thermally degrade (Beaudor et al., 2023).

Longer extraction times may increase extraction yield up to some point, but further increase in time usually cause a decrease in extraction yield. Prolonged exposure times may also result in the degradation of bioactive substances (Bhadange et al., 2022). In order to accommodate longer ultrasonic processing times, it is recommended that the pulse mode be utilized (Shen et al., 2023). This mode entails the generator intermittently activating and deactivating the power of the ultrasonic probe. By intermittently switching the power, the pulse mode effectively prevents the excessive buildup of reaction temperature during extended processing durations. Furthermore, the use of pulsed ultrasound-assisted extraction results in energy savings ranging from 20% to 51%, while enhancing the efficiency of the extraction process (Kobus et al., 2021).

The process of cavitation, in which microbubbles form and implode in the solvent as a result of ultrasonic waves, is enhanced by raising the ultrasonic power level during ultrasound-assisted extraction. Cavitation effect breaks down cell walls in the wasted coffee matrix by creating localized pressure, turbulence and heating. This shortens the amount of time needed for extraction to obtain extracts rich in bioactive compounds by accelerating the mass transfer of bioactive substances from the wasted coffee matrix to the solvent (Zupanc et al., 2019). Higher ultrasonic power levels cause cellular structures to be disrupted more effectively, thereby improving extraction efficiency. Optimal power levels are needed because high power levels usually lead to the thermal degradation of heat-labile components present in coffee waste like antioxidants, polyphenols and flavonoids (Bondam et al., 2022). Therefore, choosing the right power level is crucial to striking a balance between activity of bioactive compounds and extraction efficiency (Okur et al., 2021).

Frequency of ultrasound waves significantly impacts the cavitation effect during extraction. It affects the physical and chemical consequences resulting from disruption of cavitation bubbles during the extraction process. Different frequencies have distinct oscillation cycles. As the ultrasonic frequency declines, the oscillation cycle of cavitation bubbles rises, resulting in a larger bubble radius. This makes mechanical waves more powerful, so the cavitation effect intensifies. There is also a correlation between ultrasonic frequency and ultrasonic intensity. To achieve the desired cavitation effect, ultrasonic intensity should be amplified by increasing the frequency of ultrasonic waves to overcome the mixture's cohesiveness (Niazi et al., 2014). The energy of high frequency sound waves can be focused into a smaller region, thus providing more efficient solid and solvent interaction. This interaction could enhance microfluidic effects to extract bioactive components from the material into solution more effectively. Furthermore, ultrasonic frequency also affects the permeability of cell membrane and facilitates the passage of bioactive compounds through the cell membrane (Liu et al., 2020). The extraction efficiency of bioactive substances is also influenced by the frequency of ultrasound. The energy of the ultrasonic waves increases with increasing the ultrasonic frequency, further disintegrating the coffee grounds' cell walls. However, bioactive compounds may degrade if the ultrasonic frequency is too high (Beaudor et al., 2023). Selection of the right ultrasonic frequency is necessary to obtain bioactive compounds with the highest activity. Optimum levels of ultrasound-assisted extraction conditions of bioactive compounds for different coffee wastes are given in Table 1.

Table 1. Optimum ultrasound-assisted extraction conditions of phenolics and flavonoids for various coffee wastes

			Ultrasound-assisted extraction parameters					
Coffee Waste	Extracted Compounds	Solvent	Frequency (kHz)	Power (W)	Time (min)	Temperature (°C)	Solid Solvent Ratio	References
Coffee pulp	Total phenolics	Methanol:water	50	150	30	40	0.2:20 g/mL	Tran et al. (2020)
Coffee pulp	Total phenolics	Ethanol:water	50	250	35	60	5:100 g/mL	Tran et al. (2022)
Coffee parchment	Total phenolics	Distilled water	35	160	30	40	–	Benyelles et al. (2024)
Coffee husk	Total phenolics	Ethanol	55	60	120	20	7:210 g/mL	Andrade et al. (2012)
Coffee husk	Total phenolics, total flavonoids	Ethanol:water	40	220	60	35	1:10 g/mL	Silva et al. (2020)
Coffee silverskin	Total phenolics	Methanol:water	20	500	10	80	1:50 w/v	Wen et al. (2019)
Coffee silverskin	Total phenolics, total flavonoids	Ethanol:water	40	–	120	20	10:50 g/mL	Nzekoue et al. (2020)
Coffee silverskin	Total phenolics	Deionized water	20	60	9	20	–	Biondić Fučkar et al. (2023)
Spent espresso coffee ground	Total phenolics	Methanol:water	37	150	60	20	1:50 g/mL	Severini et al. (2017)
Spent coffee ground	Total phenolics, total flavonoids	Ethanol	20	244	34	40	1:17 g/mL	Al-Dhabi et al. (2017)
Spent coffee ground	Total phenolics, total flavonoids	Methanol:water	40	–	120	20	10:50 g/mL	Zengin et al. (2020)
Spent coffee ground	Caffeine	Distilled water	20	90	30	55	1:20 g/mL	Hassan and Al-Yaqoobi (2023)

Khochapong et al. (2021) homogenized the coffee pulp and used water extraction at the ratio of 1:2 (w/v) to extract phenolics from the coffee pulp at room temperature via solid-liquid extraction technique. The TPC recovered from the coffee pulp was found to be 11.3 mg GAE/g extract of dry weight. Solid-liquid extraction was done with hot water at 85 °C for 15 min to increase the extraction efficiency and recovery of phenolic compounds from the coffee pulp, but high temperatures lowered the TPC of coffee pulp extract to 9.2 mg GAE/g extract of dry weight (Heeger et al., 2017). Tran et al. (2022) employed the ultrasound-assisted extraction technique to extract phenolics and flavonoids from the coffee pulp, and the optimal conditions for the maximum recovery of total phenolic and flavonoid compounds yielded at a temperature of 60 °C, an ultrasonic time of 35 min and an ultrasonic power of 250 W, where the TPC and TFC reached 20.9 mg GAE/g extract of dry weight and 18.8 mg CE/g extract of dry weight, respectively. This demonstrated that, in comparison to the solid-liquid extraction method, the recovery of bioactive chemicals from the coffee pulp was nearly doubled by the ultrasound-assisted extraction method.

The phenolic and flavonoid compounds from the coffee parchment were extracted using the solid-liquid extraction technique, where the hot water at 100 °C for 90 min yielded the maximum TPC of 2.04 mg GAE/g of dry weight and the maximum TFC of 1.61 mg CE/g of dry weight (Aguilera et al., 2019). Benyelles et al. (2024) used ultrasonic-assisted extraction to recover total phenolics from coffee parchment using distilled water, where they used an ultrasonic power of 160 W, a frequency of 35 kHz, a temperature of 40 °C and an ultrasonic period of 30 min, yielding a TPC of 79.5 mg GAE/g of dry weight. These results revealed that the ultrasonic-assisted extraction considerably increased the recovery of TPC from the coffee parchment.

Hot water extraction of phenolics and flavonoids at 100 °C for a duration of 90 min from the coffee husk gave the maximum recoveries for TPC and TFC, which are 6.31 mg GAE/g of dry weight and TFC of 11.01 mg CE/g of dry weight (Rebollo-Hernanz et al., 2021). The extraction of TPC from the coffee husk in presence of ethanol was carried out using the ultrasound-assisted extraction at a power of 60 W and a frequency of 55 kHz for 120 min at 20 °C, and the TPC of 133.4 mg CGAE/g of dry weight from the coffee husk was obtained (Andrade et al., 2012). TPC of 97.9 mg CGAE/g of dry weight was extracted from the coffee husk with ethanol-water solution using ultrasound-assisted extraction, which was performed at 35 °C for 60 min at a frequency of 40 kHz and a power of 220 W (Silva et al., 2020). These findings showed that ultrasound-assisted extraction is a more powerful technique for extracting phenolic compounds from coffee husk than hot water extraction.

The solid-liquid extraction of the phenolic compounds from the coffee silverskin using different combinations of solvents (water, ethanol and aqueous solutions of ethanol), temperatures and contact times gave a maximum TPC of 16 mg GAE/g of dry weight (Costa et al., 2014). The ultrasound-assisted extraction of the phenolics from the coffee silverskin at 20 °C for 120 min at an ultrasonic frequency of 40 kHz varied with respect to solvent type and solvent concentration, where water yielded the lowest TPC of 40.4 mg GAE/g extract of dry weight while ethanol:water (70:30) solution gave the highest TPC of 73.4 mg GAE/g extract of dry weight (Nzekoue et al., 2020). In contrast, Wen et al. (2019) and Biondić Fučkar et al. (2023) found lower TPC values for the ultrasonic-assisted extraction of phenolic compounds from the coffee silverskin. This is mostly likely due to short extraction times, which do not allow complete recovery of phenolics from the coffee silverskin.

The solvent extraction of spent espresso coffee ground with ethanol-water (60:40) solution yielded a TPC of 24.25 mg GAE/g of dry weight (Zuorro, 2015). The TPC of spent coffee ranged from 19.3 to 25.5 mg GAE/g of dry weight when the spent coffee was extracted with methanol-water (60:40) solution at 60 °C for 90 min (Choi and Koh, 2017). In a similar study, the TPC values of spent coffee grounds varied between 9 and 29 mg GAE/g of dry weight depending on the coffee variety, where the solvent extraction was conducted at 60 °C for 15 min using ethanol-water (25:75) solution as the solvent (Ramón-Gonçalves et al., 2019). For the spent coffee ground extracts, the highest TPC value (587.7 mg CAE/g of dry weight) was obtained by the ultrasound-assisted extraction with ethanol as compared to the TPC value (119.5 mg CAE/g of dry weight) of the spent coffee ground extract obtained by the Soxhlet extraction (Andrade et al., 2012). An ultrasonic-assisted extraction of phenolic compounds from spent espresso coffee ground was achieved at 20 °C for 60 min using methanol-water (45:55) solution, which yielded a TPC of nearly 25 GAE/g of dry weight where the ultrasonic pulse time was less than 7.5 min (Severini et al., 2017). The low treatment temperature and short ultrasonic pulse time considerably reduced the recovery of phenolic compounds from the spent espresso coffee ground. A TPC of 36.17 mg GAE/g of dry weight and a TFC of 4.47 mg QE/g of dry weight from spent espresso coffee ground were obtained by using ultrasound-assisted extraction at an ultrasonic power of 244 W, a frequency of 20 kHz and 40 °C for the contact time of 34 min using ethanol as a solvent (Al-Dhabi et al., 2017). The ultrasound-assisted extraction of the phenolics and flavonoids from spent coffee ground at an ultrasonic frequency of 40 kHz and 20 °C for 120 min varied with respect to solvent type and solvent concentration, where methanol:water (50:50) solution yielded the highest TPC of 93.26 mg GAE/g extract of dry weight while methanol and ethanol:water (70:30) solution gave the highest TFC of 4.37 mg rutin equivalent (RE)/g extract of dry weight (Zengin et al., 2020). In a recent study, Hassan and Al-Yaqoobi (2023) compared the extraction efficiencies of ultrasonic probe and ultrasonic bath in extraction of caffeine from spent coffee ground, and determined that if the extraction time was 30 min, the ultrasonic probe was more efficient than the ultrasonic bath whereas if the extraction time increased to 60 min, the ultrasonic bath was better than the ultrasonic probe because after 30 min, the ultrasonic probe causes the degradation of caffeine.

Results showed that the ultrasound-assisted extraction ensures better recovery of phenolics and flavonoids from different coffee wastes than the solid-liquid extraction. Regarding the extraction efficiency of the ultrasound-assisted extraction on the recovery of phenolic and flavonoid compounds from various coffee wastes, studies compiled show that ultrasound-assisted extraction technique is highly efficient and beneficial method of extraction in the extraction of bioactive compounds from coffee wastes.

CONCLUSION

Coffee has a great importance and consumption rate throughout the world. The coffee industry produces significant amounts of waste, and these wastes include waste from post-harvest processing, coffee roasting, immature/defective beans, coffee husk, pulp, mucilage, parchment, silverskin and spent coffee grounds. Coffee waste is rich in phenolics and flavonoids, and extraction of bioactive compounds from coffee waste not only contributes to the recovery of valuable compounds but also reduces environmental concerns associated with high coffee waste produced by the coffee industry. Ultrasound-assisted extraction stands out as an effective extraction technique for obtaining phenolic and flavonoid compounds from coffee waste. Solvent type, solid-solvent ratio, extraction temperature, extraction time, ultrasonic power and ultrasonic frequency are important parameters influencing the ultrasound-assisted extraction of bioactive compounds. Optimizing the ultrasound-assisted extraction conditions of phenolics and flavonoids from coffee waste will provide phenolics and flavonoids with higher antioxidant activity and higher efficiency. The ultrasound-assisted extraction of phenolic compounds from coffee waste will also reduce environmental concerns associated with high coffee waste produced by the coffee industry.

CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AUTHOR CONTRIBUTIONS

Murat Özdemir contributed to the planning, writing-original draft, writing-review and editing, proofreading and supervision. Rabia Yıldırım, Rümeysa Yurttaş, Duygu Başargan and Mustafa Barış Hakcı equally contributed to the writing of the manuscript.

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