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Review

Coffee biorefinery: The main trends associated with recovering valuable compounds from solid coffee residues

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ABSTRACT

The coffee chain generates over two billion tons of solid residues annually. Husk, pulp, silverskin, defective beans, and spent coffee grounds are produced during coffee drying, husking, roasting, storage, coffee beverage preparation, and other steps. These solid materials are rich in fibers, proteins, and bioactive compounds. Therefore, this review demonstrated research trends in using solid coffee residues as a source of valuable compounds. The integral use of coffee through a biorefinery concept has shown the potential of its residues as a source of new food, pharmaceutical, materials, energy, and fertilizer products. Ultrasound, high-pressure, microwave, pulsed electric field, combining technologies, and alternative solvents (deep eutectic solvents and ionic liquids) were proposed as efficient strategies to obtain phenolic compounds, one of the highest-added-value products from solid coffee residues. However, this review verified that more standardization in reporting the studied parameters is necessary to compare the extractive methodologies. Moreover, solid coffee residues are a rich source of phenolic compounds, but researchers have reported their extraction results in total phenolic content (TPC). TPC helps to compare different extraction conditions, but chromatographic methods are much more accurate, directly identifying and quantifying each compound. Furthermore, regulatory and economic aspects regarding the use of products from coffee residues are presented. The trends indicated future studies evaluating the financial features of using emerging technologies combined with alternative solvents and more robust quantification of extracts through chromatographic techniques to obtain phenolic compounds from coffee residues.

1. Introduction

Coffee is one of the most relevant commodities in the world due to its large production worldwide. This crop is grown by over 25 million farmers in more than 60 countries (Pham et al., 2019). Moreover, coffee has been one of the most popular drinks since the mid-sixteenth century, currently associated with pleasure, health, and sustainability (Samoggia and Riedel, 2018). In addition to the flavor appreciated by consumers, coffee, as a nutraceutical, presents anti-inflammatory, antioxidant, and anti-obesity properties, helping prevent metabolic syndrome and associated disorders (Saeed et al., 2019). Furthermore, studies have shown that consumers are willing to pay more for coffee produced by a sustainable process (Samoggia and Riedel, 2018).

A sustainable process involves a series of precautions performed during the food production, process, transport, and distribution to minimize the impacts on natural resources (Wezel et al., 2020). In this sense, according to Barreto Peixoto, Silva, Oliveira, and Alves (2022), there is still a long way to attain the desired sustainability in the coffee chain, being essential to join the efforts of producers, retailers, roasters, governments, educational institutions, and organizations. Tons of solid residues are generated during the coffee chain (drying, husking, roasting, storage, coffee beverage preparation, and others). These residues include husk, pulp, silverskin, defective beans, and spent coffee grounds, constituting around 60% of the fresh fruit's wet weight, representing a relevant source of pollution and environmental threat (Alves et al., 2017; Aroufai et al., 2022; Echeverria and Nuti, 2017). According to Jiménez-Zamora et al. (2015) the coffee chain generates over two billion tons of solid residues per year, causing several problems in terms of

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disposal, environmental concerns, and specific issues associated with each type of residue. The usual destination of these residues is landfilling, despite its potential to obtain other ingredients (Arya et al., 2022).

Solid coffee residues present high value considering their content in bioactive compounds and other components that can be separated/processed to obtain new value-added co-products. Ijassi et al. (2021) clarify that solid residues from agri-food chains can be used to obtain value-added products, called co-products. In this sense, some studies have proposed a coffee biorefinery by acquiring co-products such as dietary fiber, proteins, amino acids, phenolic compounds, and others from its residues (Wen et al., 2021; Wen, Zhang, Zhao, et al., 2020; Z. Zhang et al., 2021). However, considering the high added value of phenolic compounds, most food-related research is engaged in developing efficient extractive methods for recovering these compounds from solid coffee residues.

Thus, this study aimed to demonstrate the trends related to obtaining co-products from solid coffee residues. In this sense, a brief review of solid coffee residues is provided, showing the main co-products acquired from them. A deeper examination of emerging technologies and solvents used to obtain phenolic compounds from solid coffee residues through more efficient extraction processes was presented. Finally, the main compounds (alkaloids, flavonoids, and xanthones) found in coffee waste and regulatory and economic aspects were assessed.

2. Solid residues from coffee processing and their use to obtain new products

2.1. Solid residues from coffee processing

The coffee fruit is constituted by the skin (epicarp/exocarp), the pulp (mesocarp), the parchment (endocarp), the silverskin, and two seeds, according to Fig. 1, adapted from Alves et al. (2017).

Therefore, coffee processing includes several post-harvest stages to separate the seed or bean from the other parts of the coffee fruit, preserving the final product (Alves et al., 2017). Poltronieri and Rossi (2016) reported five different methods to process coffee fruits: 1st dry process, by the use of natural or sun-dried, 2nd water-linked processing by immersion in water, 3rd pulped natural coffee by using high-tech pressure washing machines, 4th semi-dry processing-honey coffee by using depulpers and robots to remove the skin, pulp, and leaves, and 5th wet hulled/semi-washed process using de-pulping machines and de-hulling machines, and 6th wet process, where skin, pulp, and mucilage are removed using water and fermentation. However, the main ways of processing coffee are dry or wet.

The wet processing includes the steps of reception, flotation/cleaning, pulping, fermentation, washing, drying, cleaning, hulling, size grading, sorting (density/colorimetric), and storage (Vincent, 1987). Otherwise, the dry process includes the steps of reception, flotation/-cleaning, drying, hulling, size grading, sorting (density/colorimetric), and storage. The difference between the two processes is pulping, fermentation, and washing. The pulp and the mucilage are removed from the beans before the drying step in the wet method. While the dry form is more accessible and less expensive, the wet requires more equipment but ensures a much higher quality product. During the drying step performed in the dry method, many microorganisms such as molds, yeasts, and certain bacteria can increase on the coffee skin or cherries' surfaces. In the wet process, the drying of coffee fruits happens when most of the outer coverings of the coffee are removed by the pulping, fermentation, and washing steps (Vincent, 1987).

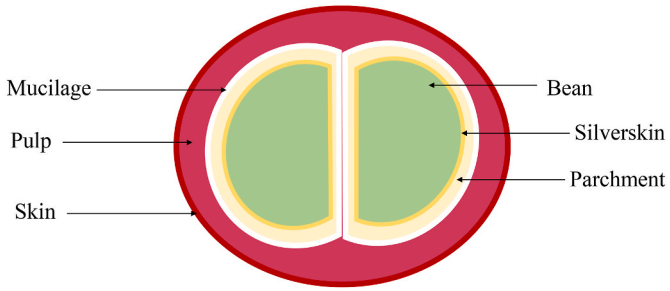

Fig. 1. Coffee fruit parts. Adapted from Alves et al. (2017).

Therefore, the solid residues generated through wet and dry processing are the skin, pulp, mucilage, and parchment of the coffee. During the dry processing method of coffee, the resulting residue is referred to as "husk," which is a composite comprising coffee skin, pulp, and parchment. (Alves et al., 2017). After the post-harvesting processes, the coffee beans were roasted. In this step, the coffee develops flavor and aroma, and another solid residue is generated, the coffee silverskin (Ruosi et al., 2012). Roasted and ground coffee beans are used to obtain soluble coffee and coffee drinks. At this stage, the waste produced is spent coffee.

Moreover, defective beans are separated from the coffee processing during the different chain steps, being another solid residue from coffee processing. Chou et al. (2019) presented the 13 classes of defective coffee beans categorized by the Specialty Coffee Association of America. Fig. 2 illustrates the solid residues produced during the coffee chain.

2.2. Solid coffee residues use

The solid residues from the coffee chain (husk, pulp, silverskin, defective beans, and spent coffee grounds) constitute around 60% of the fresh fruit's wet weight, representing a relevant source of pollution and environmental threat (Echeverria and Nuti, 2017). According to Jiménez-Zamora et al. (2015), coffee processing generates over two billion tons of solid residues per year, causing several problems in terms of disposal, environmental concerns, and specific issues associated with each type of residue. Moreover, a source of different compounds for food, pharmaceutical, materials, and energy production is wasted. Thus, studies have been conducted to obtain co-products from these residues.

Lachenmeier et al. (2022) proposed a hierarchy for using solid coffee residues, emphasizing that industries should focus on acquiring new food products (flour, distillate, tea, syrup, and beverage) from them. In sequence, materials (construction, clothing, packaging, isolation), and finally, energy (biogas, pellets, and wood) and fertilizer (compost and ashes). In addition to these products, pharmaceuticals should be explored since several studies have reported the beneficial effects of bioactive compounds obtained from coffee as anti-inflammatory, neuroprotective, antimicrobial, and anticancer (Bondam, Diolinda da Silveira, Pozzada dos Santos and Hoffmann, 2022). Table 1 presents some studies published in the last years (from 2019 to 2022) that have evaluated solid coffee residues as a source of food, pharmaceutics, material, energy, and fertilizer products.

Most recent studies have studied solid coffee residues as a source of new food or pharmaceutical products (Table 1). Moreover, most research aims to obtain bioactive compounds (phenolics, antioxidant dietary fibers, and bioactive peptides) and their activities as antioxidants for product formulation. Some studies have focused on the characterization of solid coffee residues, proposing further applications such as the one carried out by Iriondo-DeHond et al. (2019). They characterized coffee parchment, husk, and silverskin proposing their uses as food ingredients as dietary fibers and antioxidants. This study agrees with commercial interests since Bondam et al. (2022) exemplified that the AQIA company is already commercializing modified coffee fibers for application in pharmaceutical and food products.

Oil extracts acquired from green coffee beans also have been commercialized by Pectcof company (Bondam et al., 2022). In this sense, the study performed by Silva Faria, et al. (2020) demonstrated the potential of extracts acquired from green coffee beans, presenting antioxidant effects on delaying sunflower oil oxidation. Green coffee beans

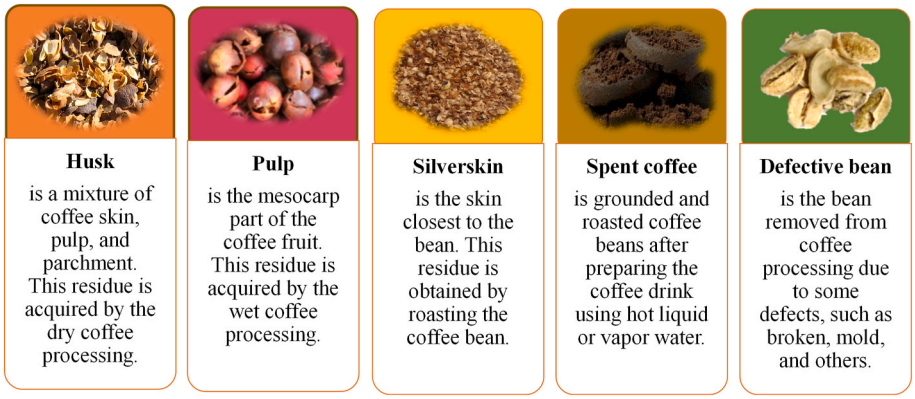

Fig. 2. The solid residues produced during the coffee chain.

are not solid coffee residues, but defective beans can be studied for this purpose since they are understudied solid residues (Table 1). Until the present review, only Prandi et al. (2021) evaluated defective beans as a source of phenolic compounds and proteins. Furthermore, food and pharmaceutical products such as brews, skin lotions, and cookies have been produced as co-products acquired from solid coffee residues. In addition to these products, different ingredients have been proposed.

Materials production using solid coffee residues have been studied mostly using spent coffee. Moreover, different materials have been proposed using coffee residues, such as a medium for yeast growth, bioactive films for packaging, adsorbents, and materials for construction. Oliveira et al. (2021) reviewed coffee residues use in plastic-based

Table 1
Co-products acquired from solid coffee residues.

Coffee residue	Proposed co-product	Reference
Food/Pharmaceutic		
Pulp	Extracts for application in innovative brews	Loukri et al. (2020)
Pulp	Antifungal extract rich in caffeic acid and epigallocatechin gallate	Sangta et al. (2021)
Pulp	Skin lotion containing antioxidant extract from coffee pulp	Widiputri et al. (2020)
Pulp	Pectin and polyphenols to use as food and pharmaceutical ingredients	Manasa et al. (2021)
Pulp	Dried coffee pulp as antioxidant dietary fiber ingredient for salty cookie formulation	Moreno et al. (2019)
Defective green beans and silverskin	Functional extracts with phenolic compounds and proteins	Prandi et al. (2021)
Husk and silverskin	Extracts with phytochemical compounds to enrich gluten-free breads formulation	Alessandro Guglielmetti, Fernandez-Gomez, Zeppa, and del Castillo (2019)
Parchment, husk, and silverskin	Antioxidant fiber, aqueous extracts enriched in phytochemicals, and antioxidant dietary fiber as food and pharmaceutical ingredients	Iriondo-DeHond et al. (2019)
Silver skin	Extracts with antioxidant and antibacterial activities	Nzekoue et al. (2020)
Silver skin	Oil with antioxidants for nutritional and cosmetics industries	del Pozo, Bartrolí, Alier, Puy, and Fàbregas (2020)
Silver skin	Proteins as food ingredient	Wen et al. (2021)
Spent coffee	Extracts with anti-proliferative and antioxidant compounds	Balzano et al. (2020)
Spent coffee	Protein hydrolysates with antioxidant and ACE (angiotensin-converting enzyme)-inhibitory activities	Valdés et al. (2020)
Spent coffee	Powder material with compounds extracted from spent coffee and whey protein with high potential for application in the dairy industry	Osorio-Arias et al. (2020)
Spent coffee	Bioactive peptides as ingredient	E. Ribeiro et al. (2019)
Spent coffee	Flour for the production of cookies	Aguilar-Raymundo, Sánchez-Páez, Gutiérrez-Salomón, and Barajas-Ramírez (2019)
Material		
Pulp	Sugar-rich hydrolysates as medium for single cell protein *saccharomyces cerevisiae*	Ubaidillah and Muzakhar (2019)
Green coffee and its residues	Bioactive films of carboxymethyl cellulose enriched with green coffee oil and its solid residues	Lombo Vidal et al. (2020)
Parchment	Extracts rich in caffeine and phenolic compounds with antifungal effects on gellan gum films as alternative in bioactive food packaging	Mirón-Mérida et al. (2019)
Parchment and spent coffee	Activated carbons as potential and low-cost adsorbents	Figueroa Campos et al. (2021)
Spent coffee	Lignins as materials for the removal of hazardous metal ions contained in metal finishing wastewater	López-Maldonado et al. (2020)
Spent coffee	Adsorbent produced with coffee residue-derived porous carbon for efficient U(VI) extraction	Chen, et al. (2022)
Spent coffee	Thermal insulation improvement in construction materials by adding spent coffee grounds	Lachheb et al. (2019)
Energy/fertilizer		
Silver skin	Biochar with potential use as an energy source	del Pozo et al. (2020)
Spent coffee	Washed hydrochar as organic amendments of an agricultural soil	Cervera-Mata et al. (2021)
Spent coffee	Biochar and crude bio-oil as sources of energy	Zhang, Yang, Wang, Rupasinghe, and He (2021)
Spent coffee	Supporting materials to produce bio-composite phase change materials with natural waxes as source of energy	Zhang, Yang, Wang, Rupasinghe, and He (2021)
Spent coffee	Bioalcohol by engineered Bacillus subtilis	Kim et al. (2020)

formulations.They proposed two distinct approaches for the valorization of coffee residues. The first approach involves utilizing crude coffee by-products as functional additives for plastics, thereby enhancing their properties. The second approach involves utilizing coffee extracts that possess film-forming abilities and functional properties, allowing them to be used as a coating material. In this sense, the antioxidant capacity of coffee compounds was also used in addition to the structuring characteristics. Otherwise, López-Maldonado et al. (2020), Figueroa Campos et al. (2021) Chen, et al. (2022) demonstrated the potential of the materials prepared with coffee residues as removing or separation agents.

The third hierarchy possibility for using coffee residues is to obtain energy (Lachenmeier et al., 2022). In this sense, Mendoza Martinez, et al. (2021) recently reviewed thermochemical routes for valorizing solid coffee residues to produce biofuels. They evaluated five technologies (gasification, torrefaction, combustion, pyrolysis, and an integrated process of torrefaction and hydrothermal carbonization) to convert the residues (pulp, husk, parchment, silverskin, and spent coffee) into heat, electricity, and biofuel energy. The coffee residues were suitable raw materials for thermochemical conversion processes. However, their water, ash, cellulose, and hemicellulose content limit the technology employed and generated energy. Some experimental studies also have demonstrated silverskin and spent coffee as sources of compounds for energy generation as biochar (Table 1). Moreover, hydrochar and bioalcohol were produced from spent coffee to amend agricultural soil and as a source of energy, respectively.

3. Emerging technologies to obtain phenolic compounds from coffee solid residues

In the last five years, many studies have been dedicated to studying coffee residues as sources of phenolic compounds due to their high added value (Table 1). Thus, different methodologies for extracting, separating, and analyzing phenolic compounds have been developed. Bondam et al. (2022) recently reviewed the extraction methods to obtain phenolic compounds from solid coffee residues. In this sense, they presented the conventional extraction techniques (agitation, heat-assisted extraction, and Soxhlet) and non-conventional (ultrasound, microwave, supercritical fluids, pulsed electric field, and fermentation) to obtain phenolic compounds from coffee residues. However, they only present the techniques and conditions that have been evaluated without discussing the advantages and disadvantages of each technology.

Conventional solid-liquid techniques, such as Soxhlet, agitation, and heated-assisted extraction, have allowed the extraction of bioactive compounds from solid coffee residues. However, they spend high solvent volume, use toxic organic solvents, and require long extraction times (Bondam et al., 2022). In this sense, emerging technologies and solvents are reviewed below to demonstrate and compare efficient processes for obtaining higher yields of phenolic compounds from solid coffee residues. Table 2 present some examples of studies that employed emerging technologies to obtain phenolic compounds from coffee residues.

3.1. High-intensity ultrasound

The high-intensity ultrasound technology is based on applying acoustic waves at high powers (>1W/cm^2) and low frequencies (1–100 MHz) in a liquid medium promoting acoustic cavitation (Strieder et al., 2021). The acoustic cavitation phenomenon starts in the medium when a value of ultrasound amplitude signal is propagated, causing compressions and subsequent decompressions of the system (Vanhille and Campos-Pozuelo, 2012). This phenomenon promotes cellular rupture facilitating the release of bioactive compounds from the raw materials to the solvent (Chemat et al., 2017). Moreover, acoustic cavitation raises mass transfer by increasing turbulence and temperature in the liquid extractive medium.

This technology has been studied in the last five years to extract phenolic, and flavonoid compounds from the coffee husk, pulp, silverskin, and spent coffee (Table 1). Although some studies reveal the highest extraction yields by using equipment that uses lower frequencies (~20 kHz), some studies still used bath-type ultrasound at a frequency of 40 kHz (Nzekoue et al., 2020; E. F. Ribeiro et al., 2019; Silva et al., 2021; Strieder et al., 2021). Moreover, there needs to be standardization of the expression of acoustic energy supplied to the system. Some studies report the nominal power, acoustic power, amplitude, or ultrasound intensity, and others do not (E. F. Ribeiro et al., 2019; Wen et al., 2019; Z. Zhang et al., 2021). In this way, as previously discussed by Strieder et al. (2019) is challenging to compare the extraction results since each research reports the ultrasound energy in one way. The most studied ultrasound variables to perform extractions are the amount of energy supplied to the system (power, amplitude, and pulses), temperature, extraction time, solvent-feed ratio (S/F), and type of solvent.

One of the first studies using ultrasound technology to extract phenolic compounds from coffee residues was performed by Severini et al. (2017). They quantified the total phenolic content (TPC) recovered from espresso spent coffee, applying ultrasound pulses at the start of the extraction, and then proceeded to a conventional solid-liquid extraction with a shaking level of 300 g^{-1}. In this sense, they realized using ultrasound pulses was essential to obtain better extraction yields. After this pulse condition, the methanol and water mixture ratio was the second most crucial parameter they studied. Usually, methanol is the best solvent for the extraction of phenolic compounds because they have a higher solubility in organic solvents than in water (Arauzo et al., 2020). Al-Dhabi et al. (2017) optimized ultrasound parameters (power, temperature, time, and solvent-feed ratio) to extract TPC, total flavonoids content (TFC), chlorogenic acid, and protocatechuic acid from spent coffee. They observed that increasing the extraction temperature from 40 to 45 °C and nominal power from 250 to 300 W promoted the thermal degradation of the extracted compounds. Moreover, a higher S/F than 25 decreased the TPC and TFC in the extract because of the reduction in the dispersion of ultrasound energy in solvent and the increment of dissolved impurities (protein, polysaccharide), which, according to them, hindered the solubilization of phenolic compounds. Arauzo et al. (2020) acquired higher yields of TPC from a previously delignified spent coffee using an ultrasound bath type. They also analyzed different solvents to perform the extraction, verifying that pure methanol provided the highest TPC yield. In this case, the lignification process increased the availability of phenolic compounds, favoring the extraction.

Otherwise, Puga, Alves, Costa, Vinha, and Oliveira (2017) proposed an environmentally more advantageous process since they used water as a solvent to extract the phenolic compounds from coffee silverskin. In this case, the highest nominal power tested by them (500 W) associated with the longest extraction time (10 min) promoted the highest extraction yield. Therefore, the acoustic cavitation promoting mechanical and thermal impacts on the extraction medium favored the extraction. Myo and Khat-udomkiri (2022) used propylene glycol as an alternative solvent to extract TPC and TFC from coffee pulp. They proposed this solvent, as polyene glycol is an ingredient in some pharmaceutical products and is generally recognized as safe (GRAS) by the U.S. A. Food and Drug Administration (FDA). Moreover, ultrasound technology associated with this innovative solvent improved the extraction yields compared with other extraction conditions, using ultrasound with ethanol, maceration with propylene glycol, and maceration with ethanol.

Some studies using high-intensity ultrasound did not report the temperature used in the extraction, which does not allow the reproduction of the experimental assay (Table 2). Many studies use room temperature to carry out the extractions. However, they should report at least the initial and final temperature of the system, considering that acoustic cavitation generates thermal energy in the medium. Solvent-to-feed ratios (S/F) from 5 to 50 have been studied for carrying out extractions, but there is a tendency and concern to reduce the amount of

Table 2
Emerging technologies to obtain phenolic compounds from coffee solid residues.

Reference	Coffee residue	Sample preparation	Technology/Equipment	Best conditions	Quantified compounds (best condition)
E. F. Ribeiro et al. (2019)	Coffee husk	Lyophilized and ground	Ultrasound bath system for cleaning	**F** (n-i), **T** (Ice bath), **S/F** (5), **t** (40 min), **solvent** (40–50% v/v ethanol	**TPC** (7.24 mg GAE/g)
Myo and Khat-udomkiri (2022)	Coffee pulp	Dried at 60 °C for 72 h and ground	6-mm ultrasound probe system	**F** (20 kHz), **ultrasound cycle** (sonication for 20 s, followed by a rest period of 20 s), **ultrasound amplitude** (40%), **T** (room temperature), **S/F** (22.22), **t** (7.65 min), **solvent** (46.71% propylene glycol solution)	**TPC** (9.29 mg GAE/g) **TFC** (58.82 mg QE/g)
Silva et al. (2021)	Coffee husk	Fresh/dried at 40 °C for 48 h and ground)	Ultrasound bath system	**F** (40 kHz), **NP** (220 W), **T** (35 °C), **S/F** (10), **t** (1 h), **solvent** (ethanol mixture 1:1 v/v)	**TPC** (90 mg CAE/g) **TFC** (15.69 mg QE/g)
Z. Zhang et al. (2021)	Coffee silverskin	Dried and ground into fractions (100–250 μm) and (500–750 μm)	1.2-cm ultrasound probe system	**F** (n-i), **NP** (49.5 W), **T** (25 °C), **S/F** (20), **t** (30 min), **solvent** (water)	**TPC** (2.79 mg GAE/g)
Puga et al. (2017)	Coffee silverskin	Ground and ungrounded	Ultrasound probe system	**F** (19.8 kHz), **NP** (500 W), **T** (room temperature), **S/F** (50), **t** (10 min), **solvent** (water)	**TPC** (20.5 mg GAE/g) **5-Caffeoylquinic acid** (0.94 mg/g)
Wen et al. (2019)	Coffee silverskin	Ungrounded	1.8-cm ultrasound probe system	**F** (20 kHz), **ultrasound intensity** (38 W/cm^2), **S/F** (50), **t** (10 min), **solvent** (methanol–water 80/20 v/v)	**TPC** (8.94 mg GAE/g) **Caffeine** (3.93 g/100 g) **3-chlorogenic acid** (17.01 μg/mL) **4-chlorogenic acid** (26.46 μg/mL) **5-chlorogenic acid** (2.78 μg/mL)
Nzekoue et al. (2020)	Coffee silverskin	Ground under nitrogen in a fine powder	Ultrasound bath system	**F** (40 kHz), **S/F** (5), **t** (120 min), **solvent** (ethanol/water 70:30 v/v)	**TPC** (8 mg GAE/g) **Caffeine** (36 mg/g) **3-chlorogenic acid** (2725 μg/g) **5-chlorogenic acid** (388 μg/g) **Caffeic acid** (212 μg/g) **Ferulic acid** (226 μg/g) **Vanillic acid** (345 μg/g)
A Guglielmetti et al. (2017)	Coffee silverskin	Ground into powder with 80 and 250 μm	Ultrasound bath system/ Microwave	***Ultrasound conditions*:** **F** (40 kHz), **NP** (300 W), **T** (66–80 °C), **t** (22 min) ***Microwave conditions*:** **Power** (280 W), **t** (32 min), **T** (51.5–80 °C) ***General conditions*:** **S/F** (35), **solvent** (60% v/v ethanol)	**Ultrasound results:** **TPC** (9.25 mg/g) **Caffeine** (14.24 mg/g) **Microwave results:** **TPC** (7.34 mg/g) **Caffeine** (11.58 mg/g)
Severini et al. (2017)	Spent espresso coffee ground	Dried at 40 °C until activity value of ~0.4	Ultrasound bath system	**F (**40 kHz**), length of ultrasound pulse** (4 min), **Pulse** (4), **T (**room temperature**), S/F** (50), **t** (60 min), **solvent** (methanol/water ratio 1.25)	**TPC** (24 mg GAE/g)
Al-Dhabi et al. (2017)	Spent coffee	Defatted and dried	2.7-mm ultrasound probe system	**F** (20 kHz), **nominal power** (244 W), **T** (40 °C), **S/F** (17), **t** (34 min), **solvent** (ethanol)	**TPC** (33.8 mg GAE/g) **TFC (**5.0 mg QE/g) **Chlorogenic acid** (1.4 mg/g) **Protocatechuic acid** (0.53 mg/g)
Arauzo et al. (2020)	Hydrochar of spent coffee	Dried at 105 °C	Ultrasound bath system	**T** (40 °C), **S/F** (25), **t** (40 min), **solvents** (water, methanol, and water:methanol)	**TPC methanol** (20,300 mg GAE/g) **TPC water** (5100 mg GAE/g) **TPC water:methanol** (19,600 mg GAE/g)
Solomakou et al. (2022)	Spent coffee	Dried at 45 °C for 24 h, freeze-dried, and defatted	13 mm ultrasound probe system/Multiwave microwave system	***Ultrasound conditions:*** **F** (20 kHz), **nominal power** (130 W), Pulse (4), **T** (60 °C), **S/F** (53), **t** (20 min), **solvent** (ethanol solution 50% v/v) ***Microwave conditions:*** **NP** (600 W), **T** (n-i), **S/F** (60), **t** (5 min), **solvent** (ethanol solution 68% v/v)	***Ultrasound result:*** **TPC** (18.52 mg GAE/g d. b.) ***Microwave result:*** **TPC** (31.8 mg GAE/g d. b.)
Shang et al. (2017)	Spent coffee	Dried in an oven at 60 °C to 15% moisture	Pressurized liquid extraction	**P (**10 MPa**), T** (95 °C), **sample loading** (0.8 g), **static time** (10 min), **solvent** (ethanol solution 70% v/v), **solvent flush** (40%)	**TPC** (22.9 mg GAE/g) **Caffeine** (9.6 mg/g)
Pettinato et al. (2020)	Spent coffee	Dried at 45 °C until constant weight	Pressurized liquid extraction	**P** (0.72 MPa), **T** (150 °C), **S/F** (10), **t** (60 min), **solvent** (ethanol 54% v/v)	**TPC** (43 mg CFAE/g)

(*continued on next page*)

Table 2 (*continued*)

Reference	Coffee residue	Sample preparation	Technology/Equipment	Best conditions	Quantified compounds (best condition)
Okur et al. (2021)	Spent coffee	Undried	Hydrostatic pressure extraction/ultrasound probe system	***High-pressure conditions*:** **P** (500 MPa), **t** (15 min) ***Ultrasound conditions*:** **F** (25 kHz), **NP** (400 W), **Amplitude** (60%), **t** (15 min) ***General conditions*:** **T** (25 °C), **S/F** (4), **solvent** (methanol solutions 10% w/v)	***High-pressure results:*** **TPC** (9.4 mg GAE/g) **Caffeic acid** (5.4 mg/g) **Chlorogenic acid** (81.2 mg/g) ***Ultrasound results:*** **TPC** (9.5 mg GAE/g) **Caffeic acid** (6.1 mg/g) **Chlorogenic acid** (85 mg/g)
Mariotti-Celis et al. (2018)	Spent coffee	Dried at 40 °C for 15 h	Pressurized liquid extraction followed by resin purification process	***High-pressure conditions*:** **P** (n-i), **T** (90 °C), **solvent** (16% of ethanol) ***Purification:*** **Solvent** (80% of ethanol)	**TPC** (8 mg GAE/g)
Pettinato et al. (2019)	Spent coffee	Dried at 323 K until constant weight	Microwave laboratory oven	**NP** (500 W), **T** (150 °C), **S/F** (10), **t** (60 min), **heating time** (10 min), **solvent** (54% v/v ethanol aqueous solution)	**TPC** (43 mg CaE/g)
Barbosa-Pereira et al. (2018)	Coffee silverskin	Ground to a powder with 0.3 mm	Pulsed electric field pre-treatment applying 12 kV, 100 A, and 50 kHz followed by solid-liquid extraction	***PEF pre-treatment*:** **Solvent** (5% v/v ethanol aqueous solution), **t** (9 μs), **number of pulses** (645), **PEF strength** (2.2 kV/cm) ***Solid-liquid extraction*:** **S/F** (640), **T** (25 °C), **ethanol concentration** (41.6%), **extraction time** (94 min).	**TPC** (13.72 mg GAE/g)
Macías-Garbett et al. (2022)	Coffee yellow and red pulp and parchment	Sun-dried (28 °C) until a constant moisture value and ground	Pulsed electric field pre-treatment applying 18 kV, and 5 Hz combined with Microwave	***PEF pre-treatment*:** **Solvent** (water), **S/F** (1), **t** (5 min), **PEF strength** (6 kV/cm) ***Microwave*:** **Solvent** (water), **S/F** (10), t (15 min)	**Yellow pulp:** **TPC** (14.3 mg GAE/g) **TFC** (3.1 mg RE/g) **Caffeic acid** (6.1 mg/g).

F: Ultrasound frequency; n-i: non-informed; T: Temperature, S/F: Solvent/feed ratio; t: Extraction time; TPC: Total phenolic content; GAE: Gallic acid equivalent; CAE: Chlorogenic acid equivalent; Ca: Caffeine equivalent; CFAE: Caffeic acid equivalent; TFC: Total flavonoid content; CE: Catechin equivalent; RE: Rutin equivalent; P: Pressure.

solvent. Often methanolic and ethanolic aqueous mixtures are employed as a solvent to perform the extractions. Thus, separating the compounds from the solvents is necessary after extraction. Therefore, reducing the solvent volume makes the separation of compounds easier.

Furthermore, water has been studied as a solvent for extractions since it is more available and environmentally friendly. The aqueous extracts can be directly applied as ingredients in formulations, reducing the costs of solvent remotion. The extraction time is another advantage of ultrasound-assisted extractions since this technology allows obtaining high yields in less time than conventional extraction techniques. The maximum extraction time studied to obtain phenolic compounds from coffee residues using the probe-type ultrasound system was 32 min. When using the bath system, longer extraction times are usually required since the dissipation of acoustic energy, or applied energy density, is lower, considering the volume of the extractor bath.

Most extraction results have been expressed in TPC and TFC, while few studies quantify the compounds by more robust methods using chromatography (Table 2). The highest TPC (20,300 mg GAE/g) acquired from solid coffee residues using high-intensity ultrasound technology was using spent coffee hydrochar as raw material (Arauzo et al., 2020). Thus, this study demonstrates that the availability of compounds in the same raw material depends on the processes it has undergone since other studies using spent coffee acquired lower yields of TPC. Moreover, Arauzo et al. (2020) obtained the best results using methanol, but water has already provided higher extraction yields observed by other studies using ultrasound technology. Furthermore, they did not evaluate the profile of these compounds, thus not demonstrating the effects of the hydrothermal delignification and extraction treatments on the extracted phenolic compounds. According to them, some phenolic compounds such as caffeic, p-coumaric, ferulic, and p-hydroxy-benzoic acid are thermally degraded at 40 °C. Therefore, if a specific phenolic compound is desired, it is necessary to optimize the extraction to obtain these compounds.

3.2. High-pressure

Technologies using high pressure to extract phytochemical compounds employ liquid solvents at high pressures and temperatures below their critical point. These temperature and pressure conditions increase the mass transfer from the matrix compounds to the extraction solvent, achieving fast and efficient extraction (Li et al., 2022). According to Khan et al. (2019), applying high pressure in a system breaks ionic bonds, decreasing the system's volume due to electrostriction forces acting on water. Thus, different phenomena happen, such as changes in reaction dynamics, and modifications in molecular structures, favoring extractions. This technology has been used in the last five years to obtain phenolic compounds from spent coffee (Table 2).

Shang et al. (2017) performed a Plackett-Burman design to choose the variables for the extraction optimization of TPC and caffeine from spent coffee. They evaluated the effects of temperature (80 and 160 °C), the concentration of ethanol solvent (25 and 75%), static time (5 and 20 min), pressure (3.4 and 17.2 MPa), the weight of the sample (0.5 and 2.5 g), and flush (20 and 100%). Thus, they observed that sample loading weight and temperature significantly affected the extraction of TPC and caffeine content. High temperatures favored the extraction by increasing the diffusion rate, mass transfer, and solubility of the compounds and the decrease of the surface tension and viscosity of the solvents. Moreover, high temperatures can promote the chemical modification of phenolic compounds, changing the bound structures. Thus, also affecting the extraction profile/yield. The sample weight otherwise affected the solvent/solid ratio (S/F), altering the diffusion rate of the extraction. In the second step of the study, they evaluated the extraction using different temperatures (40, 80, 120, and 160 °C) and sample weight (1, 1.5, 2, and 2.5 g), optimizing the results by the use of 95 °C and 0.8 g of sample. Pettinato et al. (2020) evaluated the solvent (water, ethanol, and 70% ethanol in water) to extract phenolic compounds from spent coffee. They observed that the aqueous ethanol mixture allowed

the best result of TPC. According to them, the intermediate polarity reached by mixing the most polar solvent (water) and the least one (ethanol) increased the affinity between the bioactive molecules and the liquid medium.

Mariotti-Celis et al. (2018) evaluated a combination of pressurized liquid extraction with a resin purification process to obtain phenolic compounds from spent coffee reduced in hydroxymethylfurfural compounds. According to them, hydroxymethylfurfural is a compound in spent coffee produced during the roasting process due to the Maillard reaction. They studied the remotion of this compound because of its potential human carcinogen. As observed by Shang et al. (2017) and Pettinato et al. (2020), Mariotti-Celis et al. (2018) also verified a rise in the TPC results, increasing the extraction temperature from 60 to 90 °C and ethanol concentration from 0 to 16%. Moreover, the authors observed high content of hydroxymethylfurfural in aqueous extracts. However, using ethanol-water mixtures decreased about 50% of the hydroxymethylfurfural content in the extracts due to the difference in polarity since hydroxymethylfurfural presents higher solubility in water. The best solvent to perform the desorption after the resin purification step was 80% of ethanol in water, improving the recovery of polyphenols by 20% and reducing hydroxymethylfurfural in the purified extract up to 95%. Despite this, the purification step decreased the extract's TPC content.

Okur et al. (2021) compared high-pressure technology with high-intensity ultrasound at the same temperature, S/F, and solvent-type conditions to obtain caffeic acid, chlorogenic acid, and other phenolic compounds from spent coffee. The highest pressure (500 MPa) applied for the longest time (15 min) favored the extraction by affecting the hydrophobic bonds of the cellular membranes, increasing the mass transfer rate. However, the results acquired by ultrasound technology were better using the same temperature, S/F, and processing time. In this case, high-pressure technology at high temperatures, reaching a subcritical state, could have favored the extraction of some phenolic compounds. Under these conditions, solvents present lower surface tension and greater diffusivity, which favors mass transfer and extraction of the target compounds. However, at low temperatures, acoustic cavitation by breaking the cell walls of spent coffee may have promoted the extraction of compounds more closely linked to the plant tissue.

Pettinato et al. (2020), applying a higher extraction temperature (150 °C), acquired the highest value of TPC than Shang et al. (2017) and Okur et al. (2021), which used 95 and 25 °C, respectively. However, Pettinato et al. (2020) expressed the TPC using caffeic acid as a reference, while the other studies employed gallic acid. In addition to temperature, the solvent and extraction time are essential variables considering the extraction yield. The results show that ethanolic mixtures and longer extraction times are better than methanolic solutions and shorter extraction times. However, Shang et al. (2017) should have informed the S/F ratio and the extraction time employed. As with ultrasound technology, for high-pressure technology, there needs to be more standardization in the way of expressing variables since it is difficult to compare the results of research previously conducted. Furthermore, Mariotti-Celis et al. (2018) demonstrated a different combination of extraction with a purification step that obtained purer phenolic compounds extract, reduced in hydroxymethylfurfural.

3.3. Microwave

Microwave technology promotes thermal energy inside solids containing water through electromagnetic waves applied at 915–2450 MHz (Wen, Zhang, Sun, Sivagnanam and Tiwari, 2020). The electromagnetic waves induce dipole rotation in organic molecules and heat, destroying hydrogen bonding (Akhtar et al., 2019). These microwave effects favored the mass transfer due to increases in the penetrating efficiency of the solvents into the plant matrix. Over the past five years, only some studies have evaluated the efficiency of microwave technology and its variables for extracting phenolic compounds from coffee residues (Table 2).

A Guglielmetti, D'ignoti, Ghirardello, Belviso, and Zeppa (2017) compared the technologies of microwave and ultrasound to extract phenolic compounds and caffeine from silverskin. In their study, the ultrasound technology promoted higher recovery of phenolic compounds and caffeine from coffee silverskin than the microwave. Higher temperature (80 °C) favored the extraction of caffeine by enhancing the diffusion coefficient of solvent, the solubility of the compound, the diffusion rate, and reducing solvent viscosity and surface tension. This behavior was also observed when ultrasound and conventional solid-liquid technology were used as extraction techniques to obtain phenolic compounds. However, temperatures above 51.5 °C combined with microwave waves reduced the TPC of the extract. The authors reported that phenolic compounds with several hydroxyl groups in their aromatic rings, such as caffeoylquinic acids, are unstable and can quickly degrade under microwave radiation combined with high temperatures. Otherwise, Pettinato et al. (2019) observed the opposite behavior: an increase in the extraction of polyphenols from spent coffee by increasing the microwave-assisted extraction temperature from 120 to 150 °C. According to them, this behavior implies that even if polyphenols degradation occurred, the enhancement of extraction kinetic due to temperature increase was more effective than degradation reactions. However, they applied shorter heating times (1, 10, and 20 min) than Guglielmetti et al. (2017). Moreover, the raw material source of the phytochemical was different. Furthermore, the best conditions of the microwave treatment studied by Pettinato et al. (2019) allowed the recovery of twice as many phenolic compounds in a significantly shorter time than the conventional treatment.

3.4. Combined technologies

Emerging technologies have provided higher extraction yields than conventional treatments. However, these technologies did not promote the total extraction of compounds recovered from plant raw materials. Additionally, they can expend much energy due to the time needed for extraction. Thus, the coupling of emerging technologies to extract phenolic compounds is promising since it adds different mechanisms that may favor the extraction of phenolic compounds in a shorter extraction time (Zabot et al., 2021). The extraction of phenolic compounds from coffee residues has been carried out by combining the technology of a pulsed electric field associated with solid-liquid extraction (Table 2).

3.4.1. Pulsed electric field associated with solid-liquid extraction

High-intensity pulsed electric field (PEF) technology applies pulses at high voltage intensities (10–80 kV/cm) in a medium placed between two electrodes (Toepfl et al., 2006). The pulses, applied for microseconds, promoted strong physical and chemical reactions in the medium, increasing the permeability of the membranes, causing cell inactivation, and enhancing the release of intracellular compounds, such as phytochemical compounds (Yan et al., 2017). The advantages of this technology for extracting compounds are the short operating time and the non-thermal process. Pre-treatment using electric field pulses has been applied in coffee silverskin, pulps, and parchment, favoring the sequent extraction of phenolic compounds through solid-liquid extractions (Table 2).

Barbosa-Pereira et al. (2018) studied the pre-treatment of silverskin by applying electric pulses to extract polyphenols by a solid-liquid extraction performed at 25 °C under constant rotatory agitation at 60 rpm in an orbital shaker. They evaluated the extraction efficiency through PEF treatment time (5, 9, 13, 16, 20 μs), the number of pulses (500, 645, 750, 855, and 1000), PEF strength (1.3, 2.2, 2.85, 3.5, and 4.4 kV/cm), ethanol concentration (30, 41.6, 50, 58.4, 70%), and extraction time (30, 56, 75, 94, and 120 min). The best conditions of PEF pre-treatment and solid-liquid extraction allowed an extraction yield of

19% higher in polyphenols than in control extraction (without PEF treatment). The effects that presented a significant impact on the extraction of TPC from coffee silverskin were the PEF strength, ethanol concentration, and the quadratic effects of the PEF treatment time, the number of pulses, PEF strength, and ethanol concentration.

Macías-Garbett et al. (2022) performed a pre-treatment in coffee solid residues (red and yellow pulps and silverskin) by applying a pulsed electric field pre-treatment followed by a microwave-assisted extraction. The highest TPC was obtained from yellow pulp combining the two technologies. This highest yield was followed by the red pulp and parchment results. According to the author, the two techniques promoted the cellular rupture of the vegetal tissues exposing the compounds and facilitating the mass transfer and extraction. However, the results acquired for caffeic acid recovery by combining the two technologies were statically the same achieved just by microwave extraction. The difference between TPC and caffeic acid results was attributed to the fact that PEF enhanced the extraction of other phenolic fractions more than caffeic acid. The highest caffeic acid yield value was again obtained using the combined technologies from the yellow coffee pulp (613 mg/100 g of dry sample).

3.5. Comparison between the reviewed technologies to obtain phenolic compounds

Spent coffee, silverskin, and husk are the coffee residues most studied in the last five years (from 2017 to 2022) to obtain phenolic compounds using innovative technologies (Table 2). Although some studies demonstrate the potential of green coffee beans as sources of bioactive compounds, no research was found evaluating defective beans to obtain phenolic compounds using emerging technologies (Silva Faria et al., 2020). Therefore, more studies need to be performed for processing this solid residue.

Solid coffee residues have generally been prepared for extractive processes through drying to constant moisture and grinding. Some studies have also demonstrated the effects of residue preparation on extraction yields. Silva et al. (2021) evaluated the use of fresh or dried coffee husk to obtain bioactive compounds, showing better results using the dried material. Zhang et al. (2021) verified that the extraction yield depended on the dried silverskin's particle size. They obtained the highest result using the materials with the lowest (100–250 μm) particle size. Otherwise, Puga et al. (2017) verified that unground silverskin residue allowed best extraction yields than ground material. However, in these two studies, different S/F ratios were used. Puga et al. (2017) employed a higher S/F (50) than Zhang et al. (2021) that used one of 20. A higher volume o solvent facilitated the mass transfer due to better contact between solid-liquid. In this case, at higher S/F ratio, particle size may have a lower effect on extraction results. Guglielmetti et al. (2017) observed that smaller silverskin particle sizes accelerated the extraction, allowing lower extraction times to obtain the same extraction yield.

Furthermore, Al-Dhabi et al. (2017) and Solomakou et al. (2022) defatted the spent coffee to perform the extractions. Solomakou et al. (2022) also evaluated the effects of the drying method on the extraction yield, observing that microwave technology allowed higher extraction yield using a freeze-dried sample than the dried oven sample. This difference was associated with preserving the bioactive compounds by freeze-drying and the physical changes promoted by the drying process. According to the authors, freeze-drying products present high quality due to the structural firmness on the surface where sublimation occurs, eliminating the matrix disintegration and resulting in a porous, intact structure. Despite this, the drying method did not affect the extraction yield using ultrasound technology. In this sense, the best solid coffee residue preparation method depends on the extraction technology and conditions. However, drying the material at constant moisture increases its shelf life and facilitates the penetration of the solvent into the materials, favoring extraction.

The most employed innovative technology to obtain phenolic compounds from solid coffee residues was the high-intensity ultrasound, followed by techniques using high-pressure, microwaves, and pulsed electric fields. Ultrasound technology has been studied at different frequencies (19.8–40 kHz), powers (49.5–500 W), S/F ratios (5–50), temperatures (ice bath-80 °C), processing times (7.5–120 min), and using different solvents (water, ethanol, hydroethanolic and hydro methanolic mixtures, propylene glycol solution, and DESs) (Tables 2 and 3). These extractive processes, in general, have been studied at lower temperatures compared to high-pressure and microwave technologies. This condition is an advantage considering the extraction of thermolabile compounds. However, most extraction results have been expressed in TPC. In this way, verifying the thermal degradations promoted by the extractive processes is non-possible. Pulsed electric field pretreatment also employs low temperatures, but after that, other solid-liquid extraction techniques were performed to extract the compounds. Usually, microwave techniques use shorter extraction times than those using high-pressure that employ long extraction times due to the pressurization of the systems.

The use of lower S/F ratios is an advantage, considering that after extracting the compounds, subsequent steps of separation, purification, and drying of the extracts are necessary. In this sense, the process that used the highest S/F (640) was a pulsed electric field pretreatment followed by solid-liquid extraction, and the ones that used the lowest S/F (4 and 5) used ultrasound and high pressure (Barbosa-Pereira et al., 2018; Nzekoue et al., 2020; Okur et al., 2021). Among solvents, the use of GRAS solvents is essential for further food and pharmaceutical applications. In this sense, although some studies demonstrated higher extraction yields employing methanol, its use is not recommended.

The studies using chromatographic analyses have evaluated just the extract presenting the highest TPC. However, extraction results have been primarily expressed in TPC. On the other hand, some studies demonstrated phenolic compounds' chromatographic profile but did not study the extraction conditions. In this sense, future studies should evaluate the extraction conditions and verify their effects on the phenolic compounds profile. The best TPC results have been obtained using spent coffee as a source of phenolic compounds. Furthermore, the study that allowed the highest TPC yields was performed by Arauzo et al. (2020) using high-pressure technology to extract phenolic compounds from a hydrochar of spent coffee.

4. Alternative solvents to obtain phenolic compounds from coffee solid residues

Petroleum-based solvents are the best for extracting phenolic compounds, but they present a low boiling point, are flammable, toxic, and non-biodegradable, generating a large amount of waste (Panzella et al., 2020). Thus, many studies have used water and ethanol or acidified water to extract phenolic compounds from industrial waste (Silva et al., 2021). These GRAS solvents have polar characteristics and therefore are not as efficient as other organic solvents (methanol, hexane, and others) for extracting phenolic compounds (Gallardo-Rivera et al., 2021). Thus, additional possibilities, such as using deep eutectic solvents and ionic liquids, become more interesting alternatives than water and ethanol since they have properties that can facilitate the extraction of polar and non-polar compounds. Table 3 presents some examples of studies that employed deep eutectic solvents and ionic liquids to obtain phenolic compounds from solid coffee residues.

4.1. Deep eutectic solvents

Deep eutectic solvents (DES) are a mixture of substances with a melting point at a single temperature lower than the points of the separate constituents. These solvents are formed by complexing a hydrogen bond acceptor (HBA), such as quaternary ammonium, with a hydrogen bond donor (HBD), such as urea, carboxylic acids, or amines

Table 3
Emerging solvents to obtain phenolic compounds from coffee solid residues.

Reference	Coffee residue	Alternative solvents	Best conditions	Quantified compounds (best condition)
Yoo et al. (2018)	Spent coffee	**DES: HBA** (choline chloride) **HBD** (*amine*: urea and acetamide, *alcohol*: glycerol, sorbitol, ethylene glycol, 1,4-butanediol, and 1,6-hexanediol, *acid*: malonic acid and citric acid, *sugar*: fructose, xylose, sucrose, and glucose)	**Solvent type** (1,6-hexanediol: choline chloride molar ratio 7:1), **extraction technique** (ultrasound), **t** (10 min), **T** (60 °C), **%solvent** (67.5)	**TPC** (17 mg GAE/g)
Fanali et al. (2020)	Spent coffee	**DES: HBA** (choline chloride and betaine), **HBD** (*amine*: urea, *alcohol*: glycerol, xylitol, sorbitol, ethylene glycol, 1,6-hexanediol, triethylene glycol, and propylene glycol, *acid*: citric acid and lactic acid, *sugar*: glucose)	**Solvent** (betaine and triethylene glycol (molar ratio 1:2) diluted in 30% of water (v/v), **extraction technique** (ultrasound), **T** (65 °C), **t** (20 min), **S/F** (15)	**Chlorogenic acids** (4.6 mg/g)
Ruesgas-Ramón et al. (2020)	Coffee pulp	**DES: HBA** (choline chloride and betaine), **HBD** (lactic acid, glycerol, and 1,4-butanediol)	**Solvent type** (Choline chloride and lactic acid diluted in 10% v/v of water, ratio 1:2:1.5 and ethanol 70%), **extraction technique** (heat-assisted extraction/ ultrasound), **t** (1 h/3 min), **T** (60 °C)	***DES** (heat extraction)*: **TPC** (442 mg GAE/ g) **Chlorogenic acid** (4.1 mg/g) **Caffeine** (3.9 mg/g) ***Ethanol** (ultrasound)*: **TPC** (397 mg GAE/ g) **Chlorogenic acid** (5.5 mg/g) **Caffeine** (5.9 mg/g)
Loukri et al. (2022)	Coffee pulp	**DES: HBA** (choline chloride) **HBD** (glycerol)	**Solvent type** (Choline chloride and glycerol ratio 1:3 diluted in 30% of water v/v), **extraction technique** (stirring at 600 rpm-assisted extraction), **t** (180 min), **T** (55 °C), **S/F** (47)	**Caffeine** (4.93 mg/ g)
Ferreira et al. (2021)	Spent coffee	**ILs:** Solutions of cholinium chloride ([Ch]Cl; 99 wt% pure), cholinium acetate ([Ch][Act]; 98 wt% pure), cholinium propanoate, ([Ch][Prop]; >98 wt% pure), cholinium butanoate ([Ch][But]; >98 wt% pure), cholinium dihydrogen phosphate ([Ch][DHP]; 99 wt% pure), cholinium dihydrogen citrate ([Ch][DHC]; 98 wt% pure), and cholinium bicarbonate ([Ch][Bic]; 80% in water) at 2 M.	**Solvent type** (cholinium bicarbonate ([Ch][Bic] at 1.5 M), **extraction technique** (stirring at 300 rpm-assisted extraction), **t** (30 min), **T** (80 °C), **S-L** (0.05)	**Caffeine** (3.29 wt %)

DES: Deep eutectic solvent; HBA: Hydrogen bond acceptor; HBD: Hydrogen bond donor; T: Temperature, S/F: Solvent/feed ratio; t: Extraction time; TPC: Total phenolic content; GAE: Gallic acid equivalent; S-L: Solid-liquid ratio.

(Benvenutti et al., 2019). In extracting phenolic compounds from solid coffee residues, choline chloride and betaine have been used as HBA, while different amines, alcohols, acids, and sugars have been employed as HBD (Table 3).

Yoo et al. (2018) evaluated different choline chloride-based deep eutectic solvents to extract phenolic compounds from spent coffee using ultrasound-assisted extraction. They also studied three reference solvents (water, 20% ethanol, and 30% methanol), observing that the best solvents to extract TPC and TFC from spent coffee were the DES prepared with the HBD alcohols 1,4-butanediol and 1,6-hexanediol. Moreover, the extract acquired with the DES prepared with choline chloride and 1,6-hexanediol presented the highest antioxidant activity. Therefore, this solvent was tested in different molar ratios of HBD (1, 6-hexanediol) and HBA (choline chloride), considering that this relation can alter the physicochemical properties of DESs and, therefore, the extraction efficiency. The increase of the 1,6-hexanediol ratio produced a DES with lower viscosity and allowed higher extraction yields. The molar ratio (7:1) exhibited the best results among all tested solvents, although the differences were insignificant. They employed this last DES to study other extraction parameters: DES content (50, 70, and 90% w/w), extraction time (10, 40, and 70 min), temperature (14, 42, and 70 °C), and solvent volume (mL) per 100 mg of solid (1, 1.7, and 1.4) on the extraction of phenolic compounds. First, they observed that extraction time did not affect the extraction yield, selecting 10 min. After, they optimized the extraction conditions and performed an adsorption chromatography with a macroporous resin to separate the phenolic compounds from DES. Thus, they verified that SP207 resin recovered more than 90% of extracted compounds. Ruesgas-Ramón et al. (2020) compared ethanol 70% and different formulations of DES to extract biomolecules from the coffee pulp. They used choline chloride and betaine as HBA and lactic acid, glycerol, and 1,4-butanediol as HBD to produce the DESs. All DESs were diluted in 10% (v/v) of water to obtain a more manageable viscosity to study and to maintain the superstructure characteristic of DES, which may disappear for higher amounts of added water. The best DES to extract the phenolic compounds from the coffee pulp was choline chloride and lactic acid. This solvent allowed a higher yield of TPC than the ethanolic mixture. However, the ethanol 70% mixture allowed higher chlorogenic acid and caffeine yields than the DES formed by choline chloride and lactic acid. This difference demonstrates the importance of performing chromatographic analysis since TPC is a non-specific method as the Folin-Ciocalteu can react with sugars, vitamins, organic acids, and other non-phenolic substances, leading to higher estimations. Furthermore, the authors found another peak in the chromatograms acquired for DES prepared with choline chloride and lactic acid extract. This compound was identified as furfural. According to them, furfural is formed by acid-catalyzed dehydration of pentoses and was related to hemicellulose from the spent coffee. This compound was observed in the extract acquired by long-term heating in combination with the DES. Likewise, Fanali et al. (2020) observed among the differently formulated choline chloride-based DES that the one formulated with lactic acid also presented the best efficiency for extracting phenolic compounds from spent coffee. However, except for betaine-glycerol solvent, the DES prepared with betaine showed higher extraction efficiency than choline chloride-based DES. Moreover, they diluted all their DES thirty times in water. According to the authors, the betaine characteristics are

responsible for the better results since it is an amphiphilic surfactant that can be reversibly protonated upon reducing the pH, allowing the formation of micelle-like assemblies, acting as hydrophilic and hydrophobic nanocontainers and subjecting to surface charge and size variations in dependence of pH.

Loukri et al. (2022) also demonstrated the efficiency of the DES prepared with choline chloride and glycerol to obtain caffeine from coffee pulp than other conventional solvents such as methanol and ethanol. Therefore, DES has shown more efficiency than conventional organic solvents in extracting phenolic compounds from coffee residues. The best combinations to recover the compounds from the studies already carried out are choline chloride with lactic acid and 1,6-hexanediol and betaine with triethylene glycol. Furthermore, studies have demonstrated the importance of studying the molar ratio between HBA and HBD compounds, their dilution in water, and other operational extraction parameters.

4.2. Ionic liquids

Ionic liquids (ILs) are organic salts formed by an organic cation and an organic or inorganic anion with lower melting points than their pure compounds. These have high thermal stability, negligible vapor pressure, and high hydrophobicity, so they can be reused and are considered suitable substitutes for conventional solvents (Herce-Sesa et al., 2021). We found only one study that evaluated the use of ionic liquid solutions for caffeine extraction from spent coffee, thus demonstrating a gap of opportunity for further research (Table 3).

Ferreira et al. (2021) studied seven solutions of ionic liquids to extract caffeine from spent coffee. They verified those aqueous solutions with cholinium-based ILs with butanoate and bicarbonate anions promoted higher caffeine extraction yields. According to them, this result indicated that the pH of the aqueous solutions did not impact the caffeine extraction since aqueous solutions of cholinium acetate present a pH of around 8. In contrast, cholinium dihydrogen phosphate's aqueous solutions have a pH of approximately 4. The mixture of cholinium and bicarbonate acquired the best liquid ionic solution to extract caffeine from spent coffee. This solution also had the lowest ecotoxicity among the others studied. Moreover, the temperature, IL concentration, and solid–liquid ratio promoted significant effects on caffeine extraction. As observed in the other studies presented in this review, higher temperatures (>80 °C) favored the extraction of caffeine by decreasing the IL aqueous solution viscosity, increasing mass transfer and swelling ability. Higher 1 M IL concentration also favored the extraction. Furthermore, the IL solution allowed higher extraction yields than obtained by soxhlet extraction with organic solvents (petroleum ether, n-hexane, dichloromethane, and ethanol) for 420 min. Finally, the author studied the reuse of the extract as a solvent to extract fresh raw material for six consecutive cycles. After the six cycles, the solution was not saturated with caffeine. However, the solvent lost caffeine extraction efficiency, probably due to the extraction of other compounds and solvent losses related to the solvent adsorption to the biomass. Thus, the authors verified the feasibility of solvent reuse for three cycles, with no significant losses on the caffeine extraction yield.

5. Separation and analysis method to identify and quantify phenolic compounds extracted from coffee solid residues

Most studies have determined only the total phenolic compounds' content, as observed in Table 2 (Bijla et al., 2022; Myo and Khat-udomkiri, 2022; Park et al., 2022). This analysis for comparing extraction methods is helpful. However, to quantify the compounds individually, chromatographic techniques are more suitable. Although there are many phenolic compounds to be determined, the discussion focused on the classes of alkaloids, flavonoids, and xanthones. Therefore, the three categories are discussed separately below. Fig. 3 displays the molecular structures of compounds categorized into their classes.

5.1. Alkaloids: caffeine and trigonelline

Acids and alkaloids play a crucial role in the quality of coffee, influencing the beverage's taste. Among the alkaloid molecules, caffeine and trigonelline stand out. Caffeine is a heat-stable methylxanthine and has a stimulant effect. Its content in arabica coffee beans ranges from 0.90 to 1.3% (Farah, 2012). The synthesis of trigonelline occurs by enzymatic methylation of nicotinic acid. Its degradation during roasting results in volatile compounds such as pyrroles and pyridines (Caporaso et al., 2018). Besides, this compound is not cytotoxic and acts in the prevention and treatment of caries, presenting antimicrobial action (S. A. de Almeida et al., 2021).

Several studies have been developed to quantify alkaloids (Saputri and Muchtaridi, 2018; Sousa et al., 2018; Szczesny et al., 2018; Viapiana et al., 2020). For example, Angeloni et al. (2020) reported an analytical method for simultaneously quantifying 30 compounds from spent coffee grounds. The technique used triple quadrupole HPLC with tandem mass spectrometry. The study compared extraction processes, and samples from five geographic origins were analyzed after validation of the analytical method. The average caffeine content was 1193,886 mg/kg.

The HPLC array using DAD is less sensitive than the MS/MS detector. Therefore, studies with this detector have been developed to quantitatively analyze single compounds (Angeloni et al., 2020; Nzekoue et al., 2020). For example, Zengin et al. (2020) quantified 25 bioactive compounds from spent coffee grounds and coffee silverskin by HPLC-MS/MS. Extraction with a 70:30 EtOH:H_2O ratio provided the highest caffeine content of 54,440.27 μg/g for spent coffee grounds and 41,877.13 μg/g for coffee silverskin. The high level of caffeine suggests its application in pharmaceuticals, nutraceuticals, and cosmeceuticals due to its antioxidant properties in quenching free radicals (Yashin et al., 2013). An HPLC-UV phytochemical investigation of the composition of coffee, including roots, stems, and seeds, was developed by Acidri et al. (2020). Coffee roasting reduced the total phytochemical content by 66% compared to green beans (9.70 mg/g dry weight). The heat treatment did not significantly affect the caffeine and trigonelline contents, showing 1.30 and 0.85 mg/g of dry weight for roasted beans. However, caffeine degradation was higher during senescence compared to trigonelline and other phytochemicals of the leaves.

5.2. Flavonoids: catechin, epicatechin, and naringin

There are several flavonoids in coffee residues, such as catechin, epicatechin, hyperoside, kaempferol, naringin, quercetin dihydrate, quercitrin, and others. Catechin is a polyphenol that confers antioxidant and antimicrobial activity due to orthodihydroxyl groups and hydroxyl moieties in the β ring within its molecule (Sabaghi et al., 2020). Likewise, epicatechin has several biological properties, such as antioxidant, antimicrobial, anti-inflammatory, antitumor, and cardioprotective activity (Prakash et al., 2019). Another phenolic compound in coffee by-products is naringin, which is also present in citrus fruits. Its effects include anti-inflammatory, antiallergic, antimicrobial, and anti-apoptotic properties (Zeng et al., 2021). Manasa et al. (2021) recovered pectin and polyphenols from the coffee pulp. The pectin recovery using ethanol yielded 6.7% wet weight. HPLC characterization indicated that the ethanolic extract had a catechin content of 2443 mg/100 g, while the ethyl acetate extract had an epicatechin content of 63 mg/100 g. Furthermore, the higher equivalent weight of pectin and low methoxyl content compared to commercial pectin may be due to its physical bond with polyphenols.

Hussein et al. (2022) quantified five flavonoids (apigenin-7-glycoside, naringin, chrysin, catechin, and epicatechin) by HPLC-PDA in a methanolic extract of spent coffee grounds. The catechin, epicatechin, and naringin levels were 14.55, 10.08, and 86.94 μg/g, with retention times of 11.14, 13.49, and 34.90 min. The application of the extract as a bioinsecticide against green bean pests showed a mortality between 27.5 and 76% concerning the control (7.4%) according to the concentration

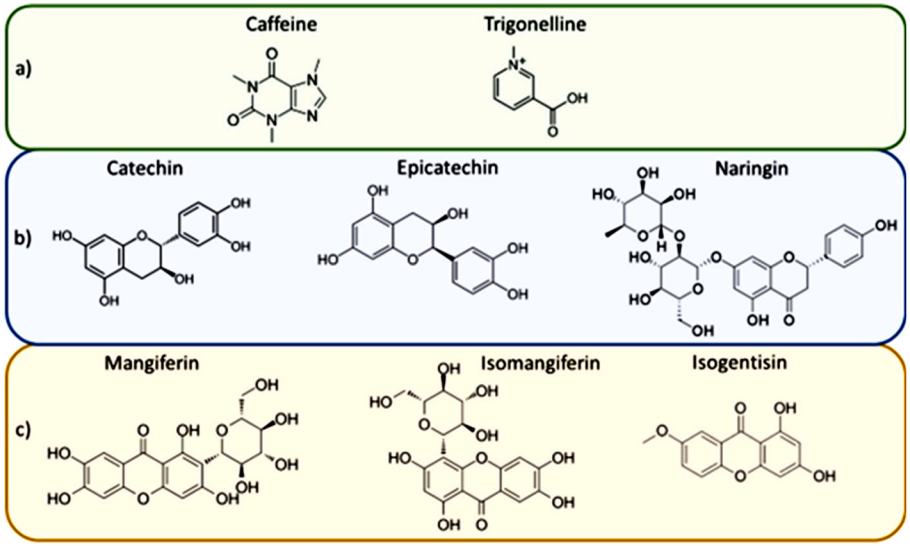

Fig. 3. Molecular structure of the main classes of polyphenols in coffee by-products: a) Alkaloids; b) Flavonoids; c) Xanthone.

of the extract. In another study, Sangta et al. (2021) evaluated the polyphenol content by HPLC, later confirmed by quadrupole time-of-flight (QTOF) liquid chromatography-mass spectrometry modes. The flavonoid with the highest content in the methanolic extract was epigallocatechin gallate (31.8 mg/g extract), followed by naringin (9.6 mg/g extract) and epicatechin gallate (8.7 mg/g extract). Antifungal activity assays showed that the acid inhibited the fungi *Paramyrothecium breviseta* with an inhibitory concentration (IC50) of 0.14. This species is a pathogen that causes leaf spots in arabica coffee.

5.3. Xanthones: mangiferin, isomangiferin, and isogentisin

Coffee leaf extract has the following xanthones in the composition: iriflophenone 3-C-glucoside, isomangiferin, mangiferin, 6-O-(p-hydroxybenzoyl)mangiferin (Cangeloni et al., 2022). In contrast, isogentisin is found in coffee silverskin (Nzekoue et al., 2020). Mangostine and mangiferin have biological activities of great pharmacological potential. Mangiferin (1,3,6,7-tetrahydroxyxanthone-C2-β-d glucoside) is a polyphenol found in many plant species, such as the Anacardiaceae and Gentianaceae families (Rajendran et al., 2014; Rodeiro et al., 2014), being present in foods. Its use in medicines aids in treating cardiovascular diseases, diabetes, cancer, and infections (Kavitha et al., 2013; Tolosa et al., 2013). Coffee leaves have an amount of mangiferin comparable to *M. indica* leaves, suggesting an excellent natural source of xanthone (Trevisan et al., 2016).

Cangeloni et al. (2022) characterized coffee leaf extracts using tandem mass spectrometry coupled with HPLC-MSn and nuclear magnetic resonance spectroscopy (NMR). The authors found four xanthone-related compounds: iriflophenone 3-C-glucoside, isomangiferin, mangiferin, and 6-O-(p-hydroxybenzoyl)mangiferin. Isomangiferin and mangiferin were eluted at a retention time of 11.63 and 12.42 min, demonstrating equal MSn fragmentation with an [M-H]- ion at m/z 421. In contrast, 6-O-(p-hydroxybenzoyl)mangiferin was eluted at a retention time of 27.10 min. Database spectra and a previous article helped the authors to identify the compounds (Malherbe et al., 2014). C-glycosylated xanthone mangiferin was identified in pulp and husks of arabica coffee varieties, although its quantification was omitted by coelution with feruloylquinic acid (Esquivel et al., 2020). Depending on the signs of mass fragmentation, the compound could be 4-O-feruloylquinic acid (Mullen et al., 2013).

Trevisan et al. (2016) compared the quantification of mangiferin and isomangiferin in methanolic extracts and infusions. Both extracts were analyzed by analytical reverse-phase HPLC electrospray ionization mass spectrometry (HPLC-ESI-MS). The mass spectra of the compounds in the negative ion mode resulted in UV spectra typical of xanthones. The base peak for both compounds was detected at [M–H]− at m/z = 421. A voltage of 100 V from the shredder provided typical C-glucoside ions. In addition, a mangiferin standard curve (0–1000 μM) from the analytical reversed-phase chromatograms at 257 nm quantified the compounds.

A method proposed by Monteiro et al. (2020) quantified mangiferin and isomangiferin in leaves of *C. eugenioides* and *C. arabica* using Ultra-HPLC-DAD. The young leaves of *C. arabica* showed total levels of xanthones (mangiferin and isomangiferin) of 15.789 mg/g dry matter. In contrast, for the young leaves of *C. eugenioides* the total levels of xanthones were 63.519 and 89.645 mg/g dry matter, depending on the coffee genotype. For the authors, the comparison of xanthones content is challenging, due to high variability, according to the geographic region of cultivation. Therefore, the environmental conditions determine the biosynthesis of secondary metabolites.

De R. F. de Almeida et al. (2019) reported different xanthones content according to the coffee-growing region in Brazil. The authors quantified the following xanthones content for coffee grown in Minas Gerais, Ceará, and São Paulo states: 8.78, 1.51, and 3.86 g/kg. The probable reason for the lower content of xanthones in the leaves from the state of Ceará was the growth in the shade, while in the other states, the growth occurred with full sun exposure. In addition to coffee leaves, coffee silverskin also contains xanthones. Contents from 0.03 to 0.50 μg/g of isogentisin were quantified in the extracts by HPLC with tandem mass spectrometry (Nzekoue et al., 2020). Isogentisin is a potent cytoprotective agent. Its action neutralized a lesion caused by smoking in the endothelial cells of the human umbilical vein (Schmieder et al., 2007). Therefore, xanthones are an important group of pharmacologically active compounds.

6. Regulatory and economic aspects regarding the use of products from coffee residues

Regulatory aspects regarding using solid coffee residues as sources of new food products were recently reviewed by Lachenmeier et al., 2021. They presented the European Union (EU) No 2015/2283 regulation that showed the status of using coffee cherry materials (husks, cascara, dried or fresh coffee cherries, and coffee pulp or mucilage), unroasted green beans, silver skin, and parchment. According to this regulation, cascara (dried coffee cherry) and cherry pulp are authorized for use since they are a traditional food in Ethiopia and Yemen countries; unroasted green beans are approved as a source of non-selective water extracts, but

selective extracts could be novel, needing authorization; the regulation for the use of silverskin is not very clear, but its consumption is carried out before 1997; finally, the parchment is a product recognized as new by regulations and is not currently approved for use.

The use of 70% aqueous ethanol extract acquired from dried whole-ground coffee fruit (including the coffee bean) in food formulations is recognized as GRAS by FDA no. 868. This authorized dried product needs to present a composition of 40% phenolic acids, with the remainder being caffeine, moisture, ash, carbohydrates, protein, and organic acids. According to this regulation, this product can be used as an antioxidant at levels ranging from 20 to 300 mg/serving in conventional foods such as flavored water/energy drink, coffee/tea, non-reconstituted protein powders beverages, milk products (prework out), clusters/bars, fruit juices, vegetable juices/blends, chocolate, candy, and chewing gum. However, according to this normative food-grade ethanol used for extraction is an unlisted GRAS substance. The ethanol used in the production meets current Food Chemical Codex (FCC) specifications. In this sense, the solvents employed for extraction, including DES and ionic liquids evaluated to extract phenolic compounds from coffee residues, need to be composed of GRAS components to be further applied in food products. No specific legislation was found to use phenolic extracts from isolated coffee residues. However, this legislation proposed by the FDA includes the use of the dried fruit as a whole, including its husk, pulp, parchment, and silver skin.

The use of phenolic extracts from coffee residues in pharmaceutical products needs to be evaluated and ensure its safety for human health, following the ICH S7A Safety pharmacology studies for human pharmaceuticals (European Medicines Agency, 2021; Amano et al., 2019). Moreover, the FDA establishes guidance for industries producing concentrated caffeine as dietary supplements, such as powdered/liquid nutritional supplements (FDA, 2018). This guidance presents the specification about the caffeine doses since it has toxic effects on the human body from a dose of 1200 mg.

The use of solid coffee residues as a source of phenolic compounds extract aiming its use as ingredients in food and pharmaceutical products present high potential from an economic and environmental point of view. These solids are low cost and usually are inappropriately discarded, causing impacts on natural resources. No studies were found evaluating the economic feasibility of processes for obtaining phenolic compounds from solid coffee residues. However, some studies have shown that the price of raw materials is one of the variables that most affect the production cost of a product (Strieder et al., 2020; Viganó et al., 2022). In this sense, using solid residues is advantageous due to their low cost. However, industries are currently focused on obtaining better quality coffee or whole grain extract for application in food products. Therefore, studies demonstrating the economic, scale-up, and environmental aspects of processes to obtain phenolic extracts from coffee residues are necessary for industrial and commercial food and pharmaceutical applications.

7. Complementary bibliometric analysis

A brief bibliometric analysis was presented, considering the numerous coffee-related publications. The methodology used for this analysis was presented as supplementary material. Fig. 4 shows the evolution of publications related to coffee co-products, with an increase from approximately 150 (2018) to over 350 (2021), placing this issue as promising for the coming years.

As verified early, coffee co-products are food, pharmaceutical, material, energy, and fertilizer products acquired from solid coffee residues (Table 1). Furthermore, Fig. 4 shows a growing interest in studies evaluating bioactive compounds' obtainment, as demonstrated in this review. Table 4 presents the rankings of the top ten countries, research areas, and journals related to coffee research. In the ranking of countries, the United States of America (USA) and Brazil are the absolute leaders in the number of publications, responsible for 18 and 16% of publications,

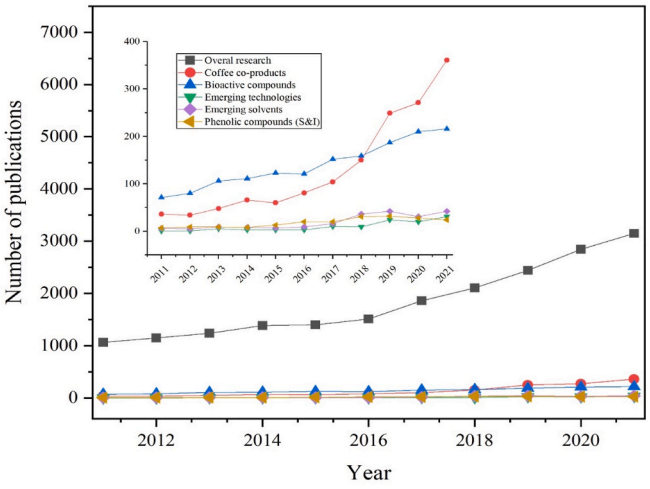

Fig. 4. Keywords associated with coffee co-products over the last ten years.

respectively. Among the publications from the USA, most of the studies are related to environmental sciences ecology, chemistry, and nutrition dietetics. At the same time, in Brazil, the most studied area is related to agriculture, followed by food science, technology, and plant sciences. This difference in the research field can be related to the country's demands. Brazilian researchers have been studying more agriculture topics, probably because the country is the world's largest coffee producer, responsible for producing 3,700,231 tons of green coffee in 2020, according to the Food and Agriculture Organization of the United Nations (2022).

The main research areas and the number of publications related to each area are also presented. Since coffee is consumed worldwide, it was expected that most of the publications would deal with the nutritional and chemical aspects of the product, passing through topics associated with its production. The journals with the most publications have an average impact factor of 5.651, ranging from 3.752 (Plos One) to 9.231 (Food Chemistry). These journals generally publish content related to food components and their bioactive constituents, food microbiology and safety, emerging technologies in food products and co-products, sustainable development, and agroecological science.

8. Final considerations

The coffee chain generates tons of solid residues such as husk, pulp, defective beans, silverskin, and spent coffee annually. These are potential sources of food and pharmaceutical ingredients, materials, fertilizers, and energetic materials. Regarding emerging technologies, we observed many studies applying acoustic energy through ultrasound devices to extract phenolic compounds from coffee residues. In addition to ultrasound, high-pressure, microwave, and pulsed electric field technologies have recently been accessed. A lack of standardization in reporting the studied variables is observed for some technologies, making it difficult to compare the best extraction results. Combining emerging technologies and alternative solvents, such as deep eutectic solvents and ionic liquids, is a trend to obtain more efficient extraction processes. Coffee extracts present a rich composition of phenolic compounds divided in this review into alkaloids, flavonoids, and xanthones. However, most studies report the extraction results in total phenolic content. This quantification allows an idea of the total compounds extracted from the material, helping to compare different extraction conditions. However, the methodology used to determine total phenolic compounds can quantify other extracted compounds, such as vitamins and sugars, presenting an overestimated result. In this sense, some studies presented in this review demonstrated that the quantification using chromatography is much more accurate, as it directly identifies

Table 4
Ranking of the 10 top publications: countries, affiliations, areas, and journals (based on 20,139 publications from the last ten years).

Criteria	Ranking	–	Number	%
Countries/Regions	1st	United State of America	3590	17.83
	2nd	Brazil	3144	15.61
	3rd	China	1779	8.83
	4th	England	1088	5.40
	5th	Germany	1088	5.40
	6th	Italy	1087	5.40
	7th	France	969	4.81
	8th	Spain	872	4.33
	9th	Japan	861	4.28
	10th	South Korea	802	3.98
Research Areas	1st	Food Science Technology	3103	15.41
	2nd	Chemistry	3001	14.90
	3rd	Agriculture	2477	12.30
	4th	Nutrition Dietetics	1768	8.78
	5th	Environmental Sciences Ecology	1765	8.76
	6th	Science Technology Other Topics	1439	7.15
	7th	Engineering	1356	6.73
	8th	Biochemistry Molecular Biology	959	4.76
	9th	Materials Science	916	4.55
	10th	Plant Sciences	893	4.43
Journals	1st	Food Chemistry	390	1.94
	2nd	Plos One	278	1.38
	3rd	Nutrients	264	1.31
	4th	Food Research International	227	1.13
	5th	Journal of Agricultural and Food Chemistry	216	1.07
	6th	Scientific Reports	165	0.82
	7th	Food Science and Technology, LWT	123	0.61
	8th	Sustainability	120	0.60
	9th	Molecules	102	0.51
	10th	Agriculture Ecosystems Environment	101	0.50

and quantifies the target compound. Thus, the trend of future research related to the topic will be associated with using emerging technologies combined with alternative solvents and more robust quantification of extracts through chromatographic techniques. Moreover, further studies should evaluate the economic viability of processes for obtaining phenolic compounds from coffee residues. Thus, sourcing valuable information for industrial and commercial purposes. The bibliometric analyses have also demonstrated the global interest in studying emerging technologies and alternative solvents to obtain co-products such as bioactive compounds from coffee residues.

Declaration of competing 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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2023.137716.

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