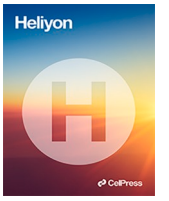
Heliyon 10 (2024) e30921

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon



Review article

Advances in the novel and green-assisted techniques for extraction of bioactive compounds from millets: A comprehensive review

Nidhi Nayak [a], Rohan Rajendraji Bhujle [b], N.A. Nanje-Gowda [c,**], Snehasis Chakraborty [d,e], Kaliramesh Siliveru [d,*], Jeyamkondan Subbiah [c], Charles Brennan [f]

[a] Department of Food Technology, Jain Deemed-to-be University, Bangalore, Karnataka, India
[b] Department of Bioscience and Bioengineering, Indian Institute of Technology, Guwahati, India
[c] Department of Food Science, University of Arkansas Division of Agriculture, AR, USA
[d] Department of Grain Science and Industry, Kansas State University, Manhattan, KS, USA
[e] Department of Food Engineering & Technology, Institute of Chemical Technology, Mumbai, India
[f] STEM College, Royal Melbourne Institute of Technology, Melbourne, Australia

ARTICLE INFO

Keywords:
Bioactive
Nutraceuticals
Green techniques
Solvent
Assisted extraction

ABSTRACT

Millets are rich in nutritional and bioactive compounds, including polyphenols and flavonoids, and have the potential to combat malnutrition and various diseases. However, extracting these bioactive compounds can be challenging, as conventional methods are energy-intensive and can lead to thermal degradation. Green-assisted techniques have emerged as promising methods for sustainable and efficient extraction. This review explores recent trends in employing green-assisted techniques for extracting bioactive compounds from millets, and potential applications in the food and pharmaceutical industries. The objective is to evaluate and comprehend the parameters involved in different extraction methods, including energy efficiency, extraction yield, and the preservation of compound quality. The potential synergies achieved by integrating multiple extraction methods, and optimizing extraction efficiency for millet applications are also discussed. Among several, Ultrasound and Microwave-assisted extraction stand out for their rapidity, although there is a need for further research in the context of minor millets. Enzyme-assisted extraction, with its low energy input and ability to handle complex matrices, holds significant potential. Pulsed electric field-assisted extraction, despite being a non-thermal approach, requires further optimization for millet-specific applications, are few highlights. The review emphasizes the importance of considering specific compound characteristics, extraction efficiency, purity requirements, and operational costs when selecting an ideal technique. Ongoing research aims to optimize novel extraction processes for millets and their byproducts, offering promising applications in the development of millet-based nutraceutical food products. Therefore, the current study benefits researchers and industries to advance extraction research and develop efficient, sustainable, and scalable techniques to extract bioactive compounds from millets.

* Corresponding author.
** Corresponding author.
E-mail addresses: nappanna@uark.edu (N.A. Nanje-Gowda), kaliramesh@ksu.edu (K. Siliveru).

https://doi.org/10.1016/j.heliyon.2024.e30921
Received 9 March 2024; Received in revised form 7 May 2024; Accepted 8 May 2024
Available online 11 May 2024
2405-8440/© 2024 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

1. Introduction

Millets are considered a dietary staple in various regions, including Africa, Asia, and India. Millets are a group of the cereal crops family *Poaceae* characterized by their small seed size [1]. They are grown in semi-arid regions where there is limited rainfall, and they can also adapt to different agro-climatic conditions [2]. They can be utilized as functional foods and nutraceuticals to support overall human diet and well-being [3,4]. There is a range of bioactive compounds abundantly found in millets which include polyphenols, flavonoids, phenolic compounds, carotenoids, alkaloids, anthocyanins, phytoestrogens, saponins, antinutritional compounds (tannins and phytic acid) and others phytochemicals [5–7]. This indicates that millets not only provide energy but also offer health-promoting substances, making them a valuable addition to a healthy diet. The seed coat of millet contains the highest concentration of bioactive compounds, while the starchy endosperm has a relatively low concentration [8,9]. The extraction of bioactive compounds is an important process in food processing [10] as it allows for the isolation and concentration of compounds offering antioxidant and anti-inflammatory activities, which can help to reduce the risk of chronic diseases such as cancer, cardiovascular disease, and diabetes [11–13].

The extraction of bioactive compounds from a source is influenced by several factors, such as the quality of the starting materials, solvent, and extraction method applied [14]. Conventional extraction methods such as maceration, hydro-distillation, and Soxhlet extraction use organic solvents, heat, and mechanical agitation to extract compounds. These techniques are extensively energy-driven and use high temperatures and prolonged extraction time, resulting in the thermal degradation of bioactive compounds [15]. It triggered the industry towards developing novel "green" extraction techniques that are more sustainable, rapid, reliable, energy-efficient, and safer for humans and the environment [16]. The typical characteristics of 'green extraction techniques' are more eco-friendly, require less energy or solvents, produce a higher yield of the desired compound with minimal thermal degradation, and permit easy separation of the solvent from the solute [17]. In the last decade, extraction techniques such as supercritical extraction, deep-eutectic solvent (DES) extraction, pressurized liquid extraction, ultrasound-assisted extraction, microwave-assisted extraction, pulsed electric field extraction, electric high-voltage discharge extraction, and high hydrostatic pressure extraction, have been employed to extract bioactive compounds using certain fluids and energy sources [15,18]. The natural progression in enhancing extraction processes involves merging contemporary techniques to create hybrid methods. These techniques are designed for continuous technological processes and scaling-up, resulting in improved efficiency, enhanced yields, reduced time, and costs, minimized solvent consumption, and selectivity of target compounds [19].

Research has demonstrated that these novel extraction techniques can potentially improve the quality of bioactive extracts obtained from millet grains. These techniques are useful in creating new nutraceutical products that can combat various age-related and lifestyle diseases. Currently, there exists a substantial literature addressing innovative extraction methods for extracting bioactive compounds from millets and their by-products. For example, recently the novel and green-assisted extraction techniques including DES assisted technique for phenolic [20] and flavonoid compounds in sorghum [21], foxtail millet [20,22], Supercritical fluid extraction of phenolic compounds in Foxtail millet [23,24] and sorghum [25–28], US assisted technique for phenolic and flavonoid compounds in sorghum [29–32] and proso millet [33], Microwave-assisted technique for 3-DXA (3-deoxyanthocyanidins) flavonoid [34]and phenolic compounds in sorghum [34–37] and kodo millet [38], enzyme assisted technique for polyphenol and flavonoid compounds in pearl millet [39,40], finger [39,41,42], sorghum [39,43], foxtail [44]and proso millet [45] and accelerated assisted technique for phenols compounds in sorghum millet [46], have been employed for the extraction of millet bioactive components. However, after an extensive review of the literature, and to the author's best knowledge, no comprehensive review articles have been identified that offer an in-depth analysis of novel extraction techniques and their potential applications in the context of millet grains. Within the scientific literature, there exists a conspicuous lacuna in the systematic examination of sustainable and efficient extraction methodologies for these bioactive constituents within millets. The novelty lies in its comprehensive exploration of advanced extraction methodologies, including thermal and non-thermal techniques, with a specialized emphasis on millet bioactive compounds. Unlike existing literature, this review provides an exhaustive compendium of the latest green extraction methods relevant to millets, bridging knowledge gaps and offering insights into pioneering methodologies that unlock the full potential of millets bioactive compounds for enhancing nutraceuticals benefits. Its emphasis on eco-friendly, energy-efficient alternatives and the potential for developing nutraceutical food products further distinguishes this work. By fostering a shift from conventional to sustainable extraction practices, this article catalyzes innovative applications in millet-based nutraceutical product development, making it a pivotal scientific resource for researchers and industry professionals alike.

In terms of scientific contributions, this article is multifaceted. First and foremost, it abates a conspicuous void within the existing millet extraction knowledge, providing both researchers and industry professionals with an exhaustive compendium of the latest green extraction techniques pertinent to millets. This elucidation is invaluable in that it offers insights into pioneering methodologies that unlock the full potential of millets as reservoirs of health-enhancing nutraceuticals. Furthermore, the review accentuates the shift from conventional, energy-intensive extraction methods towards eco-friendly, purity, low-energy alternatives. This transition is underscored by its significantly reduced environmental footprint and concomitant enhancement in yield. Lastly, this article fosters the prospect of formulating nutraceutical food products capable of mitigating age-related and chronic diseases, all while underscoring the advantages of the aforementioned green extraction techniques. In essence, this review serves as a pivotal scientific resource, meticulously bridging existing knowledge lacunae, promoting the adoption of sustainable extraction methodologies, and catalyzing innovative applications in the field of millet-based nutraceutical food product development. The objective of this article is to deliver a comprehensive discussion on bioactive compounds in millet, and novel and green extraction techniques tailored to the realm of millet extraction. The focus is to elucidate the underlying principles and specific process conditions that govern the extraction of these bioactive constituents. Additionally, the potential avenues of future research within millet bioactive extraction and the practical

application of this knowledge in the development of nutraceutical food products are elucidated.

2. Methodology

A systematic search was conducted using databases such as PubMed, Scopus, Google, and Google Scholar. The keywords or their combination used were “extraction” OR “phytochemicals ”OR“ bioactive compounds ”OR“ millets” OR “isolation” OR “polyphenols” OR “nutraceuticals” OR “assisted extraction” OR “sorghum” OR “pearl” OR “kodo” OR “finger” OR “foxtail” OR “barnyard” OR “millet husk” OR “millet barn” OR Phenols” OR “flavonoids” OR “3-DXA” OR “BHT” OR “byproduct” OR “flour” OR “sterol” OR “terpenes” OR “oils” OR “terpenoids” OR “millet waste” OR “antioxidant” OR “proso” OR “polysaccharides” OR “tannin” OR “anthocyanin” OR “carotenoids” OR “compound extraction” OR "Combination" OR ″X-ray assisted" OR "Gamma assisted" OR "Electron beam assisted" OR "Mechanochemical assisted" OR “Irradiation assisted” OR “Ultrasound assisted” OR “Enzyme assisted” OR “Supercritical fluid assisted” OR “Microwave assisted” OR “Deep Eutectic Solvent assisted” OR “Pulsed electric field assisted” and “Accelerated solvent assisted”. The search was conducted for published articles during the last six years (2018–2024) to retrieve articles about the novel and green extraction methods in millets and their byproducts. In cases where there was limited literature availability for millets, the search was extended back to 2015 for specific extraction methods (microwave, enzyme, supercritical fluid, pulsed electric field, accelerated solvent, and deep eutectic solvent) applied to millets or rice, thereby ensuring an adequate pool of relevant studies to comprehend and elucidate the effect of extraction methods on bioactive extraction.

3. An overview of bioactive compounds of millets

The bioactive compounds found in millets are phenolic compounds such as flavonoids, stilbenes, lignin, and condensed tannins [47]. The concentrations of various bioactive compounds in different varieties of millets are summarized in Table 1. For instance,

Table 1
Concentration of various bioactive compounds in different varieties of millets.

Bioactive compound	Millet variety	Concentration (mg/g)	Reference
Saponin	Foxtail	0.39	[60]
	Pearl	0.44	
Saponin	Proso	1.67	[61]
Saponin	Sorghum	0.086	[62]
Saponin	Finger	2.13–2.63 diosgenin eq	[63]
	Barnyard	8.38–10.26 diosgenin eq	
Saponin	Kodo	0.92	[64]
Saponin	Little	Absent	[65]
Tannin	Proso	15.59 TAE	[66]
	Foxtail	14.07 TAE	
Tannin	Pearl	2.3	[67]
Tannin	Sorghum	6.533 CE	[68]
Tannin	Finger	3.5	[5]
	Kodo	1.0–1.2	
	Little	3.32–3.37 CE	
Tannin	Barnyard	3.25–3.96	[63]
Flavonoids	Proso	0.284–0.978 RE	[69]
Flavonoids	Foxtail	28.1 RE 1.721–2.484 CE	[70]
	Pearl		
Flavonoids	Sorghum	1.06 to 1.18 CE	[71]
Flavonoids	Finger	0.757 RE	[72]
Flavonoids	Barnyard	29.02 RE	[70]
Flavonoids	Little	3.7 CE	[73]
Flavonoids	Kodo	87.53 mg RE	[5]
Total Phenolic Content	Proso	1.2 GAE	[74]
	Foxtail	1.7 GAE	
Total Phenolic Content	Pearl	2.64 GAE	[75]
Total Phenolic Content	Sorghum	0.166–0 0.362 GAE	[76]
Total Phenolic Content	Barnyard	1.51 GAE	[77]
Total Phenolic Content	Little	4.6 CE	[73]
Total Phenolic Content	Kodo	0.8 GAE	[78]
Anthocyanin	Sorghum	0.22 LE	[79]
Anthocyanin	Finger	58–69	[80]
Carotenoid	Proso	0.00366	[81]
	Foxtail	0.00173	
Carotenoid	Pearl	0.0028	[82]
Carotenoid	Sorghum	0.0006357	[83]
Carotenoid	Finger	0.00199	[81]
	Little	0.00078	
Carotenoid	Barnyard	0.04271	[63]

eq, equivalent; TAE, telluric acid equivalent; CE, catechin equivalent; RE, rutin equivalent; GAE, gallic acid equivalent; LE, lutein equivalent.

sorghum grain's phenolic acids include caffeic acid, p-coumaric acid, ferulic acid, sinapic acid, chlorogenic acid, protocatechuic acid, p-hydroxybenzoic acid, vanillic acid, salicylic acid, gallic acid, and syringic acid [48]. Flavonoids are abundant in the outer layers, which include anthocyanins, dihydro flavanol, flavones, flavanones, flavanols, flavanols, and isoflavones, with anthocyanins being the predominant compound [48]. Sorghum grains contain varying amounts of xanthophylls such as lutein and zeaxanthin, with lutein, zeaxanthin, and β-carotene being the most frequently found [49]. Pearl millet is characterized by a low or negligible tannin content [50], a high level of phenolic acid, and total flavonoid content [50,51], two of the flavonoids present in pearl millet are quercetin and catechin [52]. The carotenoid levels in pearl millet were significantly higher than in other millet species [52]. Additionally, C-glycosyl flavones (vitexin) were present in pearl millet [10]. Foxtail millet is another rich source of phenolics [50,51] and moderate amounts of flavonoids [51], bioactive peptides, carotenoids, and tocols. The major phenolic acids are ferulic acid, vanillic acid, and soluble forms of caffeic and sinapic acids [53]. Zeaxanthin and lutein are the most common carotenoids in foxtail. The saponins and terpenoids are present in foxtail, in addition to alkaloids, while tannins are either negligible or absent [50]. Finger millet contains ferulic acid, quercetin, and tannins. Hydroxybenzoic acids make up the major fraction of free phenolic acids in the grain. In contrast, hydroxycinnamic acids form most bound phenolic acids in finger millet [54]. Finger millet is also known to contain several flavonoids, including catechin, epicatechin, epigallocatechin, gallocatechin, and quercetin [55]. Moreover, proanthocyanidins, which are oligomeric or polymeric flavonoids, are also present [56]. Proso millet has high levels of total carotenoids, especially beta-cryptoxanthin, xanthophyll, and zeaxanthin [57]. Kodo millet, on the other hand, was found to be rich in alkyl resorcinol, coumarins, flavonoids, phenolics, and tannins [58]. Similarly, barnyard millets are rich in ortho-dihydroxy phenols, saponins, and tannins [59]. Furthermore, little millets are abundant in alkaloids, apigenin, flavonoids, luteolin, phenols, saponins, tannins, terpenoids, and kaempferol [5,50].

4. An overview of bioactives of cereals and pseudocereals

Maize is the largest cereal crop cultivated worldwide for both food and feed purposes. It contains variable concentrations of anthocyanins, with the cob containing up to four times more (3.28–3.97 g/100 g dry basis) than the grain (0.31–0.85 g/100 g dry basis). Cyanidin-3-glucoside is the predominant anthocyanin, constituting 75 % in cob and 45–47 % in grain [84]. Purple maize is rich in various phenolic acids such as ferulic, chlorogenic, and caffeic acids. Pigmented Mexican maize contains approximately 2 mg/100 g of ferulic acid, while waxy Chinese purple maize contains 0.30 g/100 g of caffeic acid [84]. Phenolic compounds like ferulic and *p*-coumaric acids are bound to non-starch polysaccharides in the cell wall [85]. The pericarp of purple maize exhibits significantly higher flavonoid concentrations (15 times that of the kernel), with kaempferol (84 %) and morin (76 %) being the major flavonoids [86]. Creole maize varieties from Brazil and China exhibit diverse total carotenoid content, with purple maize containing 0.117 mg/100 g, orange maize 0.007 mg/100 g, yellow maize 0.0108 mg/100 g, black maize 0.0086 mg/100 g, and gold maize 0.023 mg/100 g. Yellow maize is particularly rich in lutein (over 70 %), while purple and black maize contain over 90 % zeaxanthin [84].

Wheat, as the second largest cereal crop, contains various beneficial components such as anthocyanins, flavonoids, phenolic acids, tocols, carotenoids, dietary fiber, etc. The flavonoids including flavones, flavone C-glycosides, and anthocyanins, vary from 20.14 to 67.69 mg of CE/100 g. Flavonoid levels in different wheat fractions, such as bran, germ, and endosperm, also show variation ranging from 214.8 to 272.9 mg/100 g for bran or germ and 17.4–23.2 mg/100 g for endosperm [87]. Blue, purple, and red wheat whole meals exhibit different total anthocyanin levels of 150.9, 101.6, and 5.8 μg/g, respectively. Ferulic acid is the predominant phenolic acid in wheat with concentrations ranging from 269.2 to 744.7 μg/g. Wheat bran exhibits higher DF content (6.5–52.4 g/100 g) than wheat grains (11.6–17.0 g/100 g) [88]. Moreover, common, spelt, and durum wheat have an average total tocol content of approximately 5.0×10^6 μg/100 g. Carotenoids, the primary yellow pigment, include lutein, zeaxanthin, β-carotene, β-cryptoxanthin, and α-carotene, with lutein accounting for 80–90 %. Soft winter wheat bran has a total carotenoid level ranging from 130 to 180 μg/100 g [88].

Rice, the third most important cereal cultivated and consumed extensively in Asian countries, contains a range of phytochemicals and nutrients. Rice contains abundant bioactive compounds such as flavonoids (specifically anthocyanin and proanthocyanidin), carotenoids (including α−, β−carotene, lutein, and lycopene), phenolic compounds (like caffeic acid and ferulic acid), phytosterols (such as β-sitosterol, stigmasterol, and capesterol), vitamin E isoforms (α−, γ−, δ− tocotrienols and tocopherols), γ-oryzanol, coumaric acid, tricin [89,90]. The highest concentrations of bioactive compounds are typically found in the bran [91], with phenolic acids distributed across the endosperm (68 %), bran (51 %), husk (61 %), and whole grain (77 %) [89]. Tricin, among the seven reported flavonoids in rice, is predominantly present in the bran, constituting 77 % of all seven flavonoids (131.5 mg/100 g) [92]. Other flavonoids in rice include luteolin (14 %), apigenin (6 %), quercetin (3 %), isorhamnetin (1 %), myricetin (<1 %), and kaempferol (<1 %) [89]. Furthermore, rice harbors approximately 18 anthocyanins, with cyanidin-3-O-glucoside (51–84 %) being the most quantified, followed by peonidin-3-O-glucoside (6–16 %), cyanidin-3-O-rutinoside (3–5%), and cyanidin-3-O-galactoside (1–2%), showcasing significant health benefits. The mean values of these four quantified anthocyanins in pigmented rice varieties have been reported as 1252.7 and 345.8 mg/100 g in the rice bran and whole rice grain, respectively [89]. Proanthocyanidins in rice mainly consist of flavon 3-ols, such as 3-O-gallates, catechin, epicatechin, and epigallates. The husk of pigmented rice varieties is particularly abundant in catechin and epicatechin, reported with concentrations ranging from 1.57 to 3.16 and 0.57–6.14 mg/100 g, respectively [89]. The presence of α-tocopherol in rice whole grain ranges from 0.240 to 4.38 mg/100 g. The distribution of vitamin-E in rice varies in the order of husk (0.29–0.87 mg/100 g) < endosperm (1.44–1.65 mg/100 g) < whole grain (5.31–6.01 mg/100 g) < bran (24.4–24.7 mg/100 g), depending on the pigmentation phenotype of the rice grain. In whole grain, the most abundant tocotrienol out of four is γ-tocotrienol, ranging from 0.86 to 4.7 mg/100 g. γ-oryzanol varies in different parts of rice, with bran having the highest concentration (317.4–317.6 mg/100 g), followed by whole grain (41.3–47.3 mg/100 g), husk (10.2–32.3 mg/100 g), and endosperm (4.9–23.2 mg/100 g) [89].

Barley is rich in functional nutrients, such as β-glucan, arabinoxylan, and polyphenols. The aleurone layer of barley contains

β-glucan (26 %) and arabinoxylan (67 %), while the endosperm cell wall contains β-glucan (75 %) and arabinoxylan (20 %). β-glucan constitutes 22 % and 38 % of the total dietary fiber (TDF) and soluble fiber in bran, and 20 % and 27 % in flour, respectively. The Arabinoxylan contents in bran vary from 1.97 % to 8.42 %, and 0.7 %–2.13 % in flour [93]. While, Blue barley grains exhibit significant variations in phenolic compound content, including free, bound, and total phenolic acids ranging from 166.2 to 237.6, 170.1–240.8, and 336.3–453.9 mg/100 g, respectively [94]. Blue and purple barley grains have the highest flavonoid content, including potent antioxidants like oligomeric and polymeric flavan-3-ols. Significant variations exist in free (14.91–22.38 mg/100 g), bound (14.91–22.38 mg/100 g), and total flavones (37.91–47.98 mg/100 g) in 12 blue grains [93]. The predominant anthocyanin in purple barley bran is cyanidin malonyl glucoside (CMG) (9.142 g/100 g), along with cyanidin-3-galactoside pelargonidin (C3GP) (2.393 g/100 g), and cyanidin acetyl galactoside(CAG) (330.49 mg/100 g) [95]. These three anthocyanins (CMG, C3GP, CAG) constitute 98 % of the total anthocyanins in purple bran. When it comes to tocols, the endosperm holds 95 % of them, while barley hulls and germs contain about 63 % and 10 %, respectively. The tocol is also found in whole grain (5.38–12.49 mg/100 g) and pearling flour (19.5–36.3 mg/100 g), with ratios of total tocotrienols to total tocopherols approximately ranging from 1.6 to 3.9 [93].

Oat beta-glucan is the prime soluble fiber in oats, known for its cholesterol-lowering properties due to its unique viscous polysaccharide structure [96]. Additional constituents in oats are tocols, phenolic compounds, and sterols. Tocopherols and tocotrienols, collectively forming tocols, primarily constitute vitamin-E in oats [97]. The various phenolic compounds, including avenanthramides, tricin, apigenin, luteolin, kaempferol, quercetin, p-hydroxybenoic, vanillic, ferulic, caffeic, protocatechuic, syringic, p-coumaric, sinapic acids. The most abundant phenolic compounds in oat products is ferulic acid (58–78 %), followed by caffeic acid and sinapic acid. Saponins, present in rolled oats and oat porridge, demonstrate effectiveness against the growth of colon cancer cells, with reported contents of 0.9 g/kg and 0.1 g/kg (as consumed), respectively [98].

Bitter quinoa varieties exhibit saponin content ranging from 140 to 2300 mg/100 g, while in sweet it ranges from 20 to 40 mg/100 g. Quinoa's phytosterol content varies from 38.8 to 82.5 mg/100 g, with major components being β-sitosterol, campesterol, brassicasterol, and stigmasterol [99,100]. It is rich in phytoecdysteroids, ranging from 13.8 to 57.0 mg/100 g. The total polyphenol content in quinoa seeds ranges from 46. to 184 mg/100 g, with flavonol glycosides, especially quercetin and kaempferol derivatives, being the most abundant [100]. Individual phenolic acids like ferulic, caffeic, and p-coumaric acids are present, with concentrations of 25.15, 0.631, and 0.11 mg/100 g, respectively. Ferulic acid-4-glucoside is the predominant free phenolic compound, with concentrations ranging from 13.2 to 16.1 mg/100 g. Isoflavones, such as genistein and daidzein, are identified, ranging from 0.05 to 0.41 mg/100 g and 0.70–2.05 mg/100 g, respectively. Quinoa also contains tannins, with a content of 0.05 %, comparable to amaranth but higher than rice and soybean [100].

In buckwheat, bioactive compounds include flavonoids, phenolic acids, and their derivatives, triterpenoids, and stilbenes. Notably, Tartary buckwheat generally boasts a higher flavonoid content of approximately 4 g/100 g, compared to Common buckwheat's roughly 1 g/100 g. Tartary buckwheat leaves stems, and flowers are estimated to contain around 1 g/100 g of flavonoids [101,102]. Common buckwheat flowers and leaves contain 8.3–10 % and 1.2–2.6 % flavonoids, respectively [103]. The concentration of phenolic acids is highest in the brans, with *p*-hydroxybenzoic acid (up to 0.36 g/100 g fine bran), caffeic acid (38. mg/100 g fine bran), chlorogenic acid (21 mg/100 g fine bran), and protocatechuic acid (18 mg/100 g fine bran) being the most abundant [101,102]. On the other hand, protocatechuic acid is the most prevalent phenolic acid in hulls (54 mg/100 g) [101]. Buckwheat contains only one stilbene called resveratrol. Tartary buckwheat seeds contain 0.343–0.350 mg/100 g trans-resveratrol, and leaves contain 0.019–0.020 mg/100 g trans-resveratrol. In contrast, common buckwheat leaves and seeds contain 0.181–0.182 mg/100 g and 0.098–0.168 mg/100 g trans-resveratrol, respectively. Seven triterpenoids, including olean-12-en-3-ol and urs-12-an-3-ol, have been isolated from three Fagopyrum species (*F. esculentum, F. tataricum,* and *F. cymosum*). Similarly, glutinol and glutinone were identified from

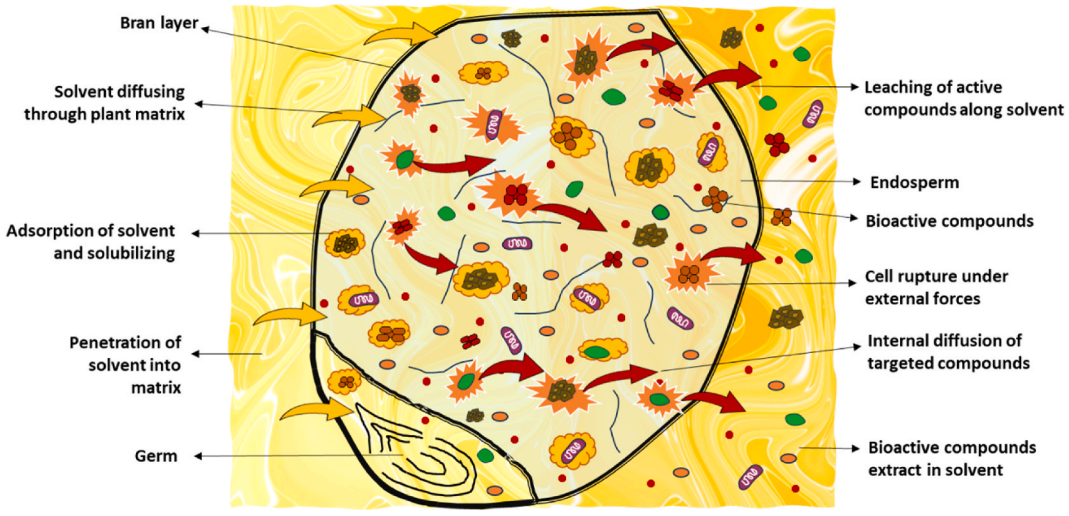


Fig. 1. Schematics of bioactive compounds in millet grains and their extraction.

F. cymosum rhizomes [102]. In fonio, the content of phenolic acids is reported as 134.42 mg/100 g of dry matter (DM), flavonoids as 3.79 mg/100 g of DM, and tannins as 34.65 mg/100 g of DM [104]. Drought-resistant amaranth accessions VA3, VA14, and VA16 showcase bioactive compounds such as polyphenols (18.425 mg GAE/100 g), ascorbic acid (158.54 mg/100 g), betaxanthins (50.54 μg/100 g), dietary fiber (7.82 g/100 g), beta-carotene (81.25 mg/100 g), flavonoids (33.55 μg RE/100 g), betacyanins (50.21 μg/100 g), total chlorophyll 77.15 mg/100 g, carotenoids 138.79 mg/100 g), and betalains (100.71 μg/100 g) [105].

5. Conventional extraction techniques and their limitations

Millets, being rich in bioactive compounds, introduce complexities in the extraction process, particularly the solubility challenge which calls for specialized methods to efficiently extract bioactive compounds [106]. Classical techniques for extracting bioactive compounds from plant materials include solid-liquid extraction (such as Soxhlet), maceration, hydro distillation, heat reflux, agitation, boiling, and leaching-out [15]. The efficiency of these conventional extraction methods primarily relies on the selection of solvents, which is dependent on the polarity of the target compound [107]. The schematic view of bioactive compounds in millets and the extraction process is shown in Fig. 1. Traditional techniques often result in low extraction yields, longer processing times, and low efficiency [15]. Additionally, the extract quality may be inferior due to residual traces of organic solvents [108]. These techniques often require mild to high temperatures (between 50 and 90 °C), thus leading to thermal degradation of the bioactive compounds [109] and oxidative degradation of sensitive constituents (e.g., reflux boiling) [110]. These methods fail to be cost-effective as they often require expensive solvents and energy-intensive techniques [109]. Furthermore, solvents exhibit low degrees of extraction selectivity, which inevitably leads to adding multiple extraction steps using different solvent steps, increasing the expenses and processing time.

Following the extraction, the solvent is evaporated, leaving behind the solutes containing the bioactive compounds. Although this step seems simple, its repercussions on the environment and human health must not be underestimated. These organic solvents exhibit a greenhouse effect and dose-dependently threaten human health [111]. The goal of extracting bioactives is for their positive impact on the health of the consumer, but the conventional methods seem to do more harm during the process of extraction alone. Therefore, there is a growing need to explore eco-friendly extraction and separation techniques. The solvents, although easy to volatilize, leave backtrace residue and are a threat to the quality of air, especially since scaling up is a part of the development process. As a result, developing green solvents and extraction techniques for millet bioactive in food applications has gained significant attention in the last

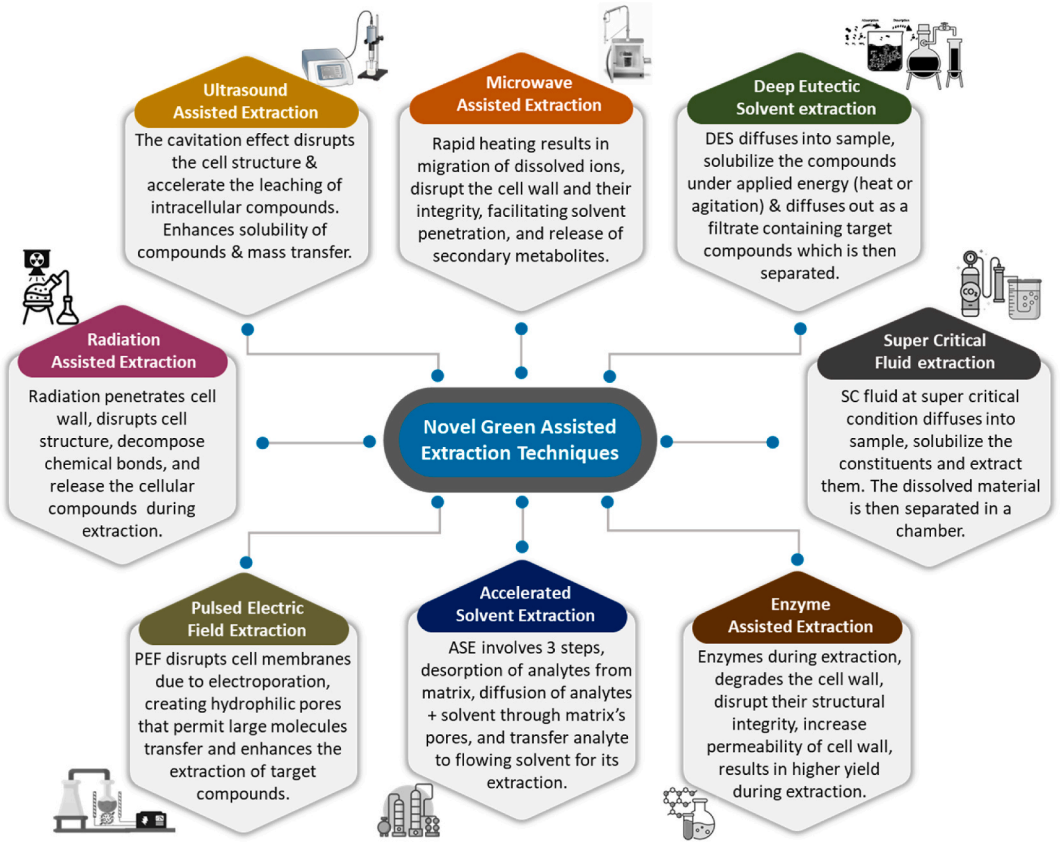


Fig. 2. Summary of the novel and green-assisted techniques for extracting bioactive compounds from millet.

decade in response to the growing demand for sustainable and environmentally friendly processes [112]. The green-assisted techniques employed in the extraction of bioactive compounds in millets and their benefits have been summarized in Fig. 2. The application of green assisted techqniues for extraction of millet bioactives has been discussed in the following sub-sections.

6. Novel and green extraction methods for millets

6.1. Ultrasound-assisted extraction

6.1.1. Principle and mechanism of extraction

The origin and progress of ultrasound can be traced back to the exploration of sound waves. The main difference lies in their frequencies: audible waves (20 Hz–20 kHz), infrasonic waves (less than 16 Hz), and ultrasonic waves (greater than 20 kHz and less than 10 MHz). Ultrasound applications are categorized into low and high intensity. Low-intensity ultrasound, with high frequency (2–10 MHz) and low power (up to 10 W), is non-invasive and widely used in the food industry [113]. High-intensity ultrasound, on the other hand, utilizes high power (100 W–10 KW) and lower frequencies (20 kHz–100 kHz) and is considered disruptive, causing mechanical and biochemical changes in liquids and gases [113,114]. Ultrasound treatment can be performed using either an ultrasonic probe or an ultrasonic bath. Probe systems are preferred for extraction due to higher power and minimal energy loss (Fig. 3).

Ultrasound assisted extraction involves creating cavitation through sound waves, disrupting the matrix structure, and enhancing the extraction process [115]. Acoustic cavitation generates shockwaves, accelerating inter-particle collisions and breaking down cellular structures. This fragmentation increases solubilization efficiency by reducing particle size and increasing the surface area and mass transfer rates [116]. Cavitation can be categorized into two types: stable and transient [117]. In stable cavitation, bubbles grow and collapse elastically over each cycle of ultrasound without collapsing at low acoustic pressure. In transient cavitation, bubbles grow rapidly and violently collapse at high acoustic pressure, resulting in extreme temperature (up to 5000 K) and pressure (up to 100 MPa) [118]. This can break down water molecules into highly reactive free radicals, such as $H+$ and OH^{-}, that can modify other molecules and accelerate certain chemical reactions [118].

6.1.2. Ultrasound-assisted extraction of bioactive compounds from millets

Ultrasonic-assisted extraction employs high-frequency sound waves to disrupt cell walls and enhance mass transfer during extraction. This mechanical action promotes the release of bioactive compounds from millet matrices, leading to increased extraction yields within shorter processing times. UAE also operates at lower temperatures compared to conventional extraction methods, minimizing the thermal degradation of sensitive compounds and preserving their bioactivity. The UAE method is considered relatively safe and sustainable, aligning with modern trends in green extraction technologies. These benefits make the UAE one of the most promising approaches for enhanced extraction of bioactive compounds. The UAE enables efficient leaching of intracellular contents, such as alkaloids, flavonoids, phenolics, phytates, and saponins [32,119], from the plant matrix into the medium, as summarized in Table 2. Lohani and Muthukumarappa [29] employed ultrasound-assisted extraction (UAE) to extract the free-bound phenolics from sorghum flour. Both batch and continuous operations were conducted while varying flour-to-water ratio (10–20 % w/v), ultrasonication intensity (30–80W/cm^2), and time (120–240 s). The amount of phenolics obtained by the continuous ultrasonication process (709 mg GAE/100 g d.m.) was higher than the batch process (656 mg GAE/100 g d.m.). This was due to the specific energy involved in the continuous method being lower (by 33 %) than the batch process. Additionally, the continuous flow process saved 35 % of processing time. Hou et al. [30] used ethanol-water solvent systems to extract phenolics and antioxidants from sorghum. The highest phenolics content of 52.23 mg GAE/g was obtained due to the high solubility of phenolics in ethanol (60 and 80 % v/v) at 50-80 °C [120]. Similarly, UAE was used with aqueous ethanol as the solvent to extract polyphenolic compounds from red sorghum bran [31]. The yield was considerably (28.7 %) higher than conventional solvent extraction (CSE). Carrera et al. [121] reported that higher

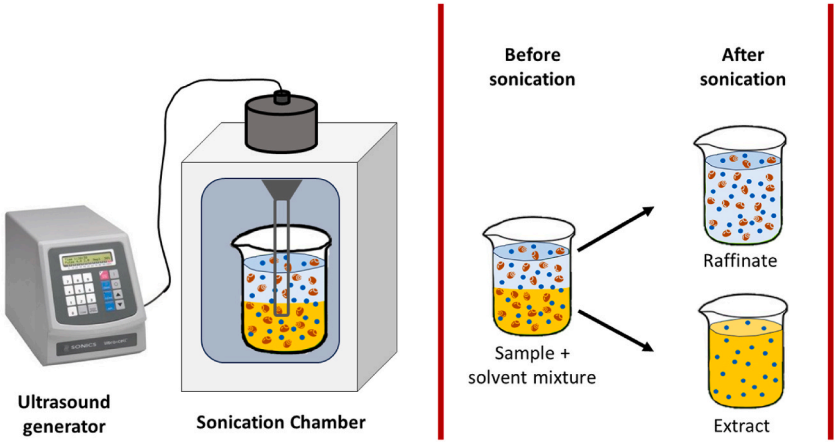


Fig. 3. Schematics showing the setup required for ultrasound-assisted extraction of bioactive compounds from millet.

Table 2
Summary of studies using ultrasound-assisted techniques for the extraction of bioactive compounds from millets.

Method	Food matrix	Process parameters	Inference	Reference
Continuous UAE	Sorghum flour	SSR: 10–30 % US intensity: 20–60 W/cm^2, Sonication time: 90–150 s Probe diameter: 22 mm	TPC yield of 7.09 g GAE/kg d.m. and AA of 1.44 mmol TE/kg d.m. at 10 % w/v SSR ratio with a UAE at 20 W/cm^2 for 130 s with a flow rate of 15 ml/s	[29]
Batch UAE	Sorghum flour	SSR: 10–20 % (w/v), US intensity: 30–80 W/cm^2, Sonication time: 120–240 s Probe diameter: 13 mm	TPC yield of 6.56g GAE/kg d.m. and AA of 1.41 mmol TE/kg d.m. at 10 % w/v SSR with an UAE at 30 W/cm^2 for 200 s	[29]
UAE	Sorghum bicolor l. Moench shells	Temperature: 40-60 °C. Solvent: 40–95 %. SSR: 1:10–1:20. US intensity: 0.24–0.40 W/cm^2 Sonication time: 10–20 min.	The highest TPC of 57.19 g GAE/kg was extracted with 80 % ethanol and 1:15 SSR at 0.32-W/cm^2 for 10 min each for two consecutive extractions.	[30]
UAE	Sorghum bran (Red)	US power: 100–300 W Sonication time: 10–30 min Ethanol concentration: 30–70 % Temperature: 30–70 °C SSR: 20–60 mL/g	The highest TPC of 49.743 g GAE/kgd.m.(28.7 % higher than CSE) was extracted with 53 % ethanol and 1:52 SSR at 200 W for 21 min.	[31]
UAE	Proso millets	US intensity: 60–100 % Sonication time: 5–20 min.	An extraction for 12.5 min at 80 % US intensity yielded a TPC of1.9 μg GAE/g, which was a 15.2 % increase over the control sample.	[122]
UAE	Sorghum sprouts	US intensity: 40–60 % Sonication time: 5–10 min	-About 14 % enhanced germination at 40 % US intensity for 5 min -The extraction for 10 min at 60 % US intensity yielded higher alkaloids, phytates, and saponins.	[32]
UAE + DES	Foxtail millet bran	DES: Betaine and Glycerol in a 1:2 ratio Water content for DES: 20–40mL/100 mL US power: 200–300 W Temperature: 50-70 °C Sonication time: 20–40 min	About 225 % higher TPC and 204 % higher TFC were extracted when treated at 247 W US power for 31 min at 61 °C, while water content for DESs was 29 mL/100 mL.	[20]

US, ultrasound; UAE, ultrasound-assisted extraction; DES, deep eutectic solvent; SSR, solid-to-solvent ratio; TPC, total phenolic content; TFC, total flavonoid content.

temperatures lead to higher recovery rates in solid-liquid extractions. However, the high-temperature conditions also promoted the oxidation degradation of phenolic compounds.

Ultrasonication can speed up the process of soaking and germination of stored seeds [123,124]. Hassan et al. [32] reported that the sonication at 40 % amplitude for 5 min increased the germination rate of sorghum sprouts from 80 % to 94 %. Additionally, the alkaloids, phytates, and saponins in the untreated sample were increased from 0.051 to 0.062, 146–154.4, and 0.15–0.17 mg/100 g, respectively, after sonication at 60 % amplitude for 10 min. The quality and nutritional profile of the grains are enhanced due to the breakdown of complex substances into simpler compounds during germination. These simpler compounds are transformed into essential nutrients that can be more easily absorbed by the developing plant embryo, resulting in variations in bioactive compounds. The high-intensity ultrasound pretreatment can also improve the nutritional quality of millet bran by converting its insoluble/bound dietary fiber and antioxidant compounds into a soluble/free form, making them more available for absorption by the body. Mustac et al. [122] employed ultrasound pretreatment to increase the freely available bioactive in proso millets. A soaking of 15 % millet bran in water and sonication at 80 % amplitude (400 W/cm^2) for 12.5 min resulted in a 15.2 % higher total phenolic content and 16.3 % improved antioxidant activity. Raj et al. [125] suggested that sonication enhances the TPC yield because of high-intensity ultrasound's effect on polyphenol oxidase (deactivation), leading to less breakdown of polyphenols, resulting in higher yields. These studies confirm that ultrasonication emerges as a potent green extraction method for millet bioactive compounds, showcasing its efficacy in leaching diverse constituents from cereal flour. Continuous ultrasonication proves superior, yielding higher phenolic content with reduced energy consumption and processing time. Solvent variations, including ethanol-water systems, elevate phenolic yields, surpassing conventional methods. Ultrasonication not only facilitates extraction but also accelerates seed soaking and germination, enhancing millet's nutritional profile. High-intensity ultrasound pretreatment transforms insoluble compounds in millet bran, improving their bioavailability. Future research should optimize ultrasonication parameters, explore nutritional changes during germination, investigate enzyme interactions, devise scalable strategies, and conduct comparative studies for a comprehensive understanding of ultrasonication's environmental impact and energy efficiency. These endeavors may advance the application of ultrasonication in green

extraction techniques, addressing both efficiency and sustainability concerns.

6.1.3. Combination of ultrasound and deep eutectic solvent

A promising new type of solvent that is gaining attention is called deep eutectic solvent (DES). DES has been applied in combination with other techniques in recent days to extract bioactive like alkaloids, cannabinoids, ginkgolides, polyphenols, etc. [112]. Compared to traditional solvent extraction, DES-based UAE produced higher levels of total phenolics, flavonoids, in vitro antioxidant activity, and acetylcholinesterase inhibitory activity. Ultrasound assisted extraction (UAE) using DES was carried out to extract phenolic compounds from foxtail millet bran (FMB) [20]. A mixture of betaine and glycerol, in a ratio of 1:2, was chosen based on its ability to extract the most phenolic compounds. The total phenolic content (TPC) of 7.80 mg ferulic acid equivalents (FAE)/g and total flavonoid content of 8.89 mg rutin equivalents (RE)/g were twice the yield obtained using CSE [54]. FMB treated with UAE-DES showed more pores and cracks on the surface than CSE-treated grains. Wang et al. [126] correlated the TPC extraction efficiency with the DES water content, which was increased from 10 mL/100 mL–30 mL/100 mL. This may be attributed to the weakening of the hydrogen bonds, which reduce the viscosity of the DESs, thus facilitating the extraction of TPC. The phenolic extract from FMB using DES-based UAE contained fifteen main phenolic compounds, with p-coumaric acid, apigenin-C-dihexoside, and N′,N″-di-p-coumaroylspermidine being predominant. Noteworthy that 1-O-p-coumaroylglycerol was detected in FMB for the first time [11].

Understanding the mechanisms of UAE, the interaction between waves and components, and its effect on quality extracted compounds is crucial for its effective utilization in food processing. There is limited research on ultrasound-assisted extraction in millets, particularly in minor millets. Further research is needed to explore the potential of the UAE in comprehending the effect of ultrasound on biochemical constituents and their extraction from millets. The integration of Deep Eutectic Solvents (DES) with Ultrasonic-Assisted Extraction (UAE) emerges as a promising strategy for enhancing millet bioactive extraction. UAE-DES exhibits notable advantages, surpassing traditional methods by yielding higher phenolic and flavonoid contents in foxtail millet bran. The morphological changes observed in grains post-UAE-DES treatment highlight its potential for modifying millet matrix characteristics, a crucial aspect in bioactive extraction. However, a critical research gap exists, particularly in minor millets, necessitating in-depth exploration of the underlying mechanisms of UAE-DES interactions. The optimization of UAE parameters, along with an expanded application spectrum in millet processing, holds tremendous potential for future advancements in green extraction techniques, ensuring efficient and sustainable utilization of millet bio actives for diverse applications, including functional foods and nutraceuticals. The advantages of UAE include enhanced mass transfer, increased extraction yields, and reduced thermal degradation due to lower operating temperatures. UAE is relatively safe, and sustainable, and aligns with green extraction principles by minimizing solvent usage and energy consumption. However, drawbacks such as equipment costs, potential cavitation-induced sample damage, and the need for optimization to achieve maximum efficiency must be considered. Precautions involve optimizing parameters like ultrasonication intensity, duration, and solvent composition to prevent over-processing or sample degradation. Additionally, safety measures to mitigate exposure to high-frequency sound waves and adherence to standard operating procedures are essential. A thorough understanding of UAE's mechanisms, coupled with careful parameter optimization and safety protocols, ensures its effective and responsible application in bioactive compound extraction from millets.

6.2. Microwave-assisted extraction

6.2.1. Principle and mechanism of extraction

Microwaves are a non-ionizing electromagnetic wave that falls within the frequency range of 300 MHz to 300 GHz [127]. Domestic and industrial microwave systems utilize 2450 MHz microwaves with a wavelength of 0.122 m, while large-scale industrial systems use 915 MHz microwaves with a higher penetration depth and wavelength of 0.327 m [128]. Microwave induces heat through ionic conduction and dipole rotation mechanisms [129]. Ionic conduction involves ion movement in an electromagnetic field, generating heat in polar solvents. Dipole rotation causes random molecular movement and heat generation, particularly in polar solute molecules [130]. Both mechanisms often occur in unison under microwave radiation of frequency 2450 MHz, which causes dipoles to align and randomize at 4.9×10^9 Hz [131]. Dielectric materials with permanent dipoles attempt to align with the electric field, but friction from rapid changes leads to molecular vibration and heat generation [131].

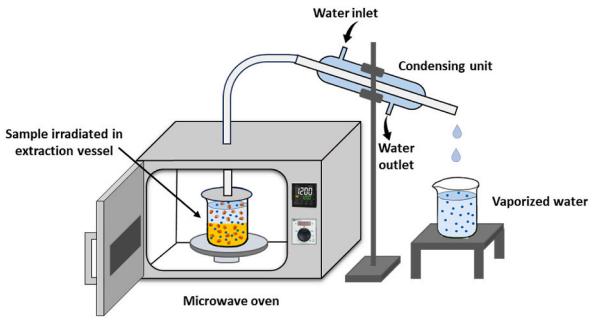


Fig. 4. A schematic showing the setup required for microwave-assisted extraction of bioactive compounds from millet.

Microwave assisted extraction (MAE) is a method that rapidly delivers energy to a mixture of solvent and solid matrix, causing both to heat up almost instantaneously. Additionally, the migration of dissolved ions facilitates solvent penetration into the matrix, resulting in increased chemical release. Closed microwave systems are suitable for extreme conditions [132] and consist of a magnetron tube, an oven with rotating platforms for extraction vessels, temperature and pressure monitoring equipment, and electronic components (Fig. 4). The process involves adding the sample and solvent and sealing the vessel. Microwave radiation heats the solvent in a short pre-extraction step (<2 min), followed by irradiation and extraction for 10–30 min. Open-system extractions are conducted at atmospheric pressure, limiting the maximum temperature to the boiling point of the solvent [133].

6.2.2. *Microwave-assisted extraction of bioactive compounds from millets*

Microwaves are also considered green-assisted techniques as they do not use harmful chemicals or solvents for extraction. The method is also known as solvent-free microwave extraction. Microwaves can also be integrated with conventional solvent or vacuum microwave-assisted techniques that use no solvents or green solvents such as water, ethanol, and methanol acetone to enhance extraction efficiency, reduce processing time, and increase the yield of compounds [134]. The integration of vacuum offers a benefit and alternate option to perform the extraction of heat-sensitive compounds at lower temperatures. Microwave assisted extraction (MAE) has proven itself to be a booming technology for the isolation of phenolic compounds from millet's bran matrix [135]. Table 3 summarizes the studies using microwave-assisted techniques for the extraction of bioactive compounds from grains. Duke et al. [37] subjected sorghum bran to MAE in acidified methanol and microwaved for 10 min between 300 and 1200 W. The MAE yield was significantly higher (3–32 times) than the conventional solvent extraction (CSE). MAE enhanced the extractability via deglycosylation and depolymerization reactions. On the other hand, MAE is one of the least effective methods for extracting pigments such as anthocyanins from plant tissue [136]. It is theorized that the degradation of anthocyanins through nucleophilic attack leading to hydration at C-2 is a major mechanism induced by MAE. Herrman et al. [35] employed microwave radiation (1–30 min at 300–1200 W) to ground sorghum grains in a 10:1 solvent (1 % HCl in methanol (v/v%) to sample ratio. The yield increased 2 folds compared to CSE, which might be attributed to thermal stability and the resistance of the heterocyclic ring to cleavage of 3-DXA [35].

MAE over traditional alkaline refluxing offers several benefits, including higher extraction efficiency, accelerated processing, and reduced solvent usage [138]. Chiremba et al. [36] investigated the relationship between grain hardness and bound phenolic acids and found no correlation. However, extraction of sorghum-bound phenolic acids was done for 90 s in 2 M NaOH, which displayed

Table 3
Summary of studies using microwave-assisted techniques for the extraction of bioactive compounds from millets.

Method	Matrix	Process parameters	Inference	References
MAE	Sorghum grains	Power: 300–1200 W Time: 1–30 min SSR: 1:10	The sample treated for 30 min at 600 W yielded about 3.1 mg/g of 3-DXA (twice the CSE yield)	[35]
MAE	Sorghum grains	Power:400 W Time: 15–90 s SSR: 1:25 Solvent: 2 M NaOH	Phenolic acids were extracted faster after 45 s at 400 W	[79]
MAE	Sorghum bran	Power:300–1200 W Time: 10 min SSR: 1:40 Solvent: 1 % HCl in methanol	-Phenolic yield, antioxidant capacity, andhydroxycinnamic acids increased by > 3 times after 10 min extraction at 1200 W	[37]
UAE and MAE	Sorghum hulls	UAE: US intensity: 916–1120 W/cm^2 Time:20–30 min Solvent: 60 % ethanol Particle size: 100–500 μm MAE: Power: 50–70 W Time:90–120 s Solvent: 60 % ethanol Particle size: 100–500 μm	-MAE (63 W/101 s with 100 μm) offered the highest TFC of 195 mg CE/g dw (13 % higher than UAE) -UAE for 23.6 min at 1078 W/cm^2 yielded TPC of 779.0 mg GAE/g d.w (29 % higher than MAE) when the particle size was 100 μm	[34]
US-MAE	Sorghum husk	Power: 14–33W Time: 10–30min US intensity: 7.45 W/cm^2 Pulses: 2s ON and OFF Temperature: 45–65 °C SSR: 1:30 Solvent: 7:3 ethanol and water (1 % HCl v/v)	3-DXA pigment yield was 3.6 times higher than CSE after 20 min of extraction	[137]
MAE	Kodo millet husk	Time: 2–6 min Temperature: 60–100 °C Solvent: 30–90 % ethanol in water	Treatment for 5.81 min at 100 °C with 49.8 % ethanol v/v extracted 175.24 μg ferulic acid equivalents/g (17 times higher than CSE)	[38]

CSE, conventional solvent extraction; MAE, microwave-assistedextraction; SSR, solid-to-solvent ratio; TPC, total phenolic content; UAE, ultrasound-assisted extraction; US, ultrasound.

outstanding improvement in processing duration. The drastic improvement in the processing rate must be credited to the ether bonds, heat-labile at 170 °C, while the reaction mixture was at 190 °C [36]. The waste generated during millet processing majorly comprises husk, which has been utilized in the production of bioethanol [139]. However, extracting phenols and other bioactive before those uses could be a potent valorization technique. Palaniswamy [38] investigated the valorization of kodo millet husk by extracting phenols and other antioxidants by MAE. The finalized protocol suggested that yields of phenols were significantly greater compared to CSE [140].

Microwave-Assisted Extraction (MAE) has demonstrated remarkable efficacy in extracting bioactive compounds from millets, particularly phenolic compounds from bran matrices. Studies reveal significantly enhanced yields compared to conventional methods, attributed to MAE's ability to induce deglycosylation and depolymerization reactions. However, challenges persist, such as the reduced effectiveness in extracting certain pigments like anthocyanins due to potential degradation mechanisms. The exploration of MAE's benefits, including accelerated processing and reduced solvent usage, positions it as a superior alternative to traditional methods, offering valuable insights into the relationship between grain characteristics and bound phenolic acids. Future implications include optimizing MAE parameters for diverse millet varieties, addressing pigment extraction challenges, exploring valorization techniques for millet waste, aligning with sustainable practices, and advancing the extraction efficiency of bioactive compounds for various applications in the food and pharmaceutical industries.

6.2.3. Microwave in combination with other techniques

UAE and MAE are becoming more common in the extraction industry. A comparison of UAE and MAE in extracting phenolic compounds from discarded sorghum hulls was carried out [34]. MAE was more efficient in extracting anthocyanin (28.2 %), flavonoids (13.2 %), and red pigments (22.5 %), while UAE extracted higher total phenols (28.6 %). Flavonoids showed the highest yield under MAE at higher exposure times as they increased the contact time between solute and solvent but started decomposing at microwave power beyond 60W [141]. In the case of TPC, a higher yield was obtained by the UAE. A higher sonication power intensity promoted cell wall disruption, facilitating solvent access to the cell content, and intensifying the mass transfer rate. At lower particle size, the contact surface area of the solute and solvent was higher, thus increasing the mass transfer rate [137].

Sorghum husk was subjected to ultrasound-microwave-assisted extraction in 70 % acidified ethanol while varying temperature and time at ultrasound intensity of 7.45W/cm^2 and microwave intensity between 14 and 33W [142]. The yield of pigment molecules mainly containing 3-DXA was enhanced by 3.6 folds compared to CSE [35]. This is credited to the combination of ultrasound and microwave in a single reactor, where ultrasound facilitates the solubilization of bioactive components in solvents due to reduced particle size, increased surface area, and higher mass transfer rates in the solid matrix boundary layer [143,144]. The microwave causes rapid heating while decreasing the viscosity of solvents and improving penetration into the matrix, thereby improving the extraction efficiency [142]. While numerous studies have investigated MAE on a small scale in laboratories, future research should focus on upscaling the extraction process from millets. To enhance the performance of MAE, process intensification techniques can be explored. These techniques involve integrating microwave heating with other extraction methods such as ultrasound, pressurized techniques, or enzyme-assisted extraction. Combining different techniques can achieve synergistic effects, leading to improved extraction efficiency.

The combination of MAE and UAE has emerged as a potent strategy for extracting bioactive compounds from sorghum hulls and husks. Comparative studies reveal that MAE excels in extracting specific compounds like anthocyanins and flavonoids, while UAE demonstrates superiority in total phenol extraction. The synergistic effects of ultrasound and microwave in a single extraction process enhance solubilization and improve mass transfer rates. Future implications include upscaling MAE from millets, exploring process intensification techniques, and leveraging synergies with other extraction methods for enhanced efficiency, opening avenues for large-scale applications in the food and pharmaceutical industries. Microwave assisted extraction (MAE) presents several merits but also requires scrutiny regarding its advantages, drawbacks, and necessary precautions. Benefits include rapid heating and energy delivery, increased extraction efficiency, and reduced solvent usage, making it a green-assisted technique. MAE's ability to induce deglycosylation and depolymerization reactions enhances bioactive compound yields, particularly phenolic compounds from millet matrices. However, drawbacks such as reduced effectiveness in extracting certain pigments like anthocyanins due to potential degradation mechanisms must be considered. Precautions involve optimizing parameters like microwave power and duration to prevent excessive heating, and degradation of heat-sensitive compounds like flavonoids and the sample itself. A comprehensive understanding of MAE's mechanisms, coupled with careful parameter optimization and safety protocols for handling microwave radiation, ensures its effective and responsible application in bioactive compound extraction from millets.

6.3. Deep eutectic solvents extraction

6.3.1. Principle and mechanism of extraction

Mixtures with a melting point lower than the ideal eutectic temperature are called deep eutectic solvents (DES's). These solvents typically have freezing points that fall within the range of −69 °C to –149 °C, with all of them having a freezing point below 150 °C [145]. However, DES is employed in liquid form at room temperature or below 70 °C [146]. Unlike ionic liquids, which are often expensive, non-biodegradable, and can have high toxicity, DES's are typically inexpensive, biodegradable, non-toxic, and easy to prepare [146]. Natural DES comprises organic acids, amino acids, sugars, polyols, and choline derivatives, all primary metabolites [147]. Its components are found in daily diet, such as choline, citric acid, betaine, amino acids, and sugars [148]. Natural DES is generally recognized as safe by the FDA, and the resulting extracts can be directly added to food products to improve their nutritional value.

An appropriate DES is selected based on the specific target compounds, considering the solubility requirements (Fig. 5). The DES is then prepared by thoroughly mixing the selected components in the desired ratio, typically through heating and stirring to ensure proper dissolution [149]. After which, the raw material containing the bioactive compounds undergoes preparation, which may include grinding [150], drying, or other pre-treatment methods [22,151]. The prepared sample is mixed with the DES, and selected extraction techniques are applied. Following extraction, the DES mixture is separated from the solid residue [20] using filtration or centrifugation techniques. This separation step yields a filtrate that contains the bioactive compounds dissolved in the DES. The subsequent recovery stage involves further processing to isolate and purify the desired bioactive compounds [152].

6.3.2. *Application of DES-assisted technique in the extraction of bioactive compounds*

Eutectic solvents offer a unique advantage by providing a tailored solvent environment that can effectively dissolve specific bioactive compounds. These solvents are composed of natural, biodegradable components, making them environmentally friendly alternatives to traditional organic solvents. DES can be customized to match the polarity and solubility requirements of target compounds, thereby improving extraction efficiency and selectivity. The DES method is considered relatively safe and sustainable, aligning with modern trends in green extraction technologies. DES is affordable, environmentally friendly, safe, and straightforward to create. It is also recognized as safe by the FDA, these DES extracts can be seamlessly integrated into food items to enhance their nutritional profile. Its ability to enhance extraction efficiency while maintaining the integrity of bioactive compounds makes it a promising option for researchers and industries focused on extracting valuable components from millets for various applications in food, pharmaceuticals, and nutraceuticals. However, the application of DES to extract bioactive compounds from millets is still in its infancy stage. There are few studies demonstrating this technique in extraction (Table 4). The use of DES for lignocellulosic biomass pretreatment is relatively new; DES can improve enzymatic hydrolysis by facilitating the dissolution of lignin from cellulosic biomass [153]. Das et al. [21], extracted lignin from sorghum biomass using DES followed by catalytic transfer hydrogenolysis (CTH), which produced lower molecular weight phenolic compounds. Under optimal conditions (270 °C, reaction time of 60 min with 15 % Ru/C catalyst), about 36.3 % of lignin oil yield was recorded.

6.3.3. *Combination of DES and other assisting extraction techniques*

Zhang et al. [150] utilized 6-di-*tert*-butyl-4-methylphenol (BHT) and additional phenolic antioxidants to impede oxidation of the β-carotene from millet powder, coupled with different extraction techniques to optimize the yield. The finalized protocol (0.25g of millet powder in 1 mL DES with grinding-assisted extraction) achieved 91 % of total β-carotene yield, 3-fold higher than ethanol extract. The enhanced yields were attributed to the DES facilitating better contact between the sample and extraction solvents, resulting in efficient extraction of β-carotene. Effective extraction of bioactive compounds relies heavily on the pretreatment of the sample. High hydrostatic pressure (HHP) is a trending process technology that utilizes pressure between 100 and 1000 MPa. This process results in the rupture of cells and the formation or damage of non-covalent bonds such as hydrogen, hydrophobic, and ionic bonds [154]. Zhang et al. [154] subjected millets to HHP pretreatment followed by DES extraction. The optimized protocols were 500 MPa for 15 min, followed by DES extraction with octanoic acid + linalool at a 1:1 M ratio, with a solid-to-liquid ratio of 1:10 g/mL for 80 s. This recorded polyphenol yields 1.5–3 folds greater than CSE. HHP treatment disrupts the salt bridges and deprotonates the charge groups and hydrophobic bonds in the cell membrane, which results in higher permeability. The decrease in the dielectric constant of water also plays a role in enhancing bioactive compound content by reducing the bipolar properties of the media [155]. The

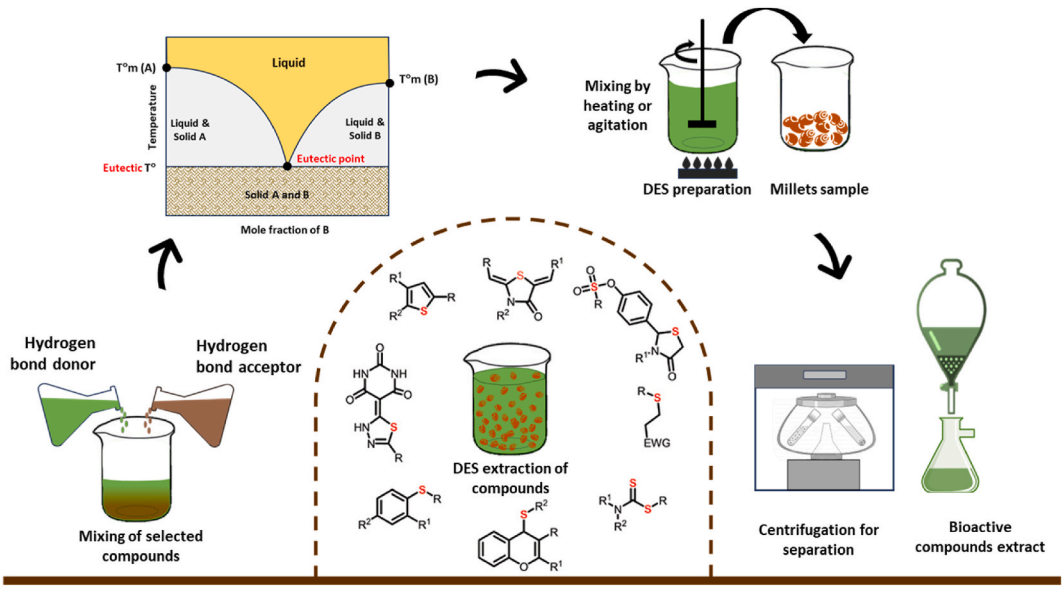


Fig. 5. A schematic showing the principle of deep eutectic solvent-assisted extraction of bioactive compounds from millet.

Table 4
Summary of studies using deep eutectic solvent (DES) assisted techniques for the extraction of bioactive compounds from millets.

Method	Matrix	Process parameters	Inference	Reference
DES	*Sorghum bicolor* biomass	Solvent: Choline chloride: Lactic acid = 1:2 M ratio SSR: 1:9 RPM: 200 Temperature: 270 °C Time: 0.5, 1, and 3 h Catalyst: 0,2, 5, 15 and 20 Ru/C	Lignin oil content of 36.3 % and 2.63 Ru/C of phenol were obtained at reaction conditions of 270 °C for 1h with 15 % Ru/C catalyst.	[21]
DES + GAE/ WBE/ UAE	Unknown millet variety	DES: N,N-dimethylcyclohexylamine/N,N-dimethyloctylamine/N,Ndimethylbenzylamine + n-butanol molar ratio: 6:1, 5:1, 4:1, 3:1, 2:1, 1:1, 1:2, and 1:3 Phenolic Antioxidants: 2,6-di-*tert*-butyl-4-methylphenol (BHT), 2(3)-*tert*-butyl-4-methoxyphenol (BHA), and 2-*tert*-butyl1,4-benzenediol (TBHQ) at0.1, 1, 2, 3, 4, 10, and 15 mg/mL SSR:0.1–0.5 g/mL Time: 5–60s Extraction Methods: GAE, WBE, and UAE Switch: NaOH (Hydrophobic) or HCl (Hydrophilic) DES reuse: 1–6 times	-The highest yield was observed when 0.25 g/mL of millet powder in DES (N,N-dimethylcyclohexylamine + n-butanol at a molar ratio of 3:1) with 3 mg/mL of BHT when used along with GAE. −About 91 % recovery was achieved after DES was used 5 times with hydrophilic and hydrophobic transformation each time.	[149]
HHP + DES	Unknown millet variety	HHP: Millet + water1:1 Pressure: 300–600 MPa Time: 5–20 min. DES: Octanoic/Nonanoic/Decanoic acid + Linalool molar ratio: 3: 1, 2: 1, 1: 1, 1: 2, and 1: 3 β-Cyclodextrin concentration: 10–30 mg/mL SSR: 1:50–1:5 g/mL Time: 20–120 s	-The optimal extraction was obtained at 500 MPa for 15 min, followed by DES extraction with octanoic acid + linalool at a 1:1 M ratio, with SSR of 1:10 g/mL for 80 s with β- cyclodextrin concentration of 16 mg/mL. -The antioxidant activity of DES extract was significantly higher (1.5–3 times) than that of CSE with ethanol as solvent.	[153]
UAE + DES and UAE	Foxtail millet husk	DES: Betaine/L-lactic acid/Sodium acetate + Glycerol/ Glycol/L-lactic acid molar ratio of 1: 4, and 1: 2 Water in DES: 10–20 % SSR: 0.1 g/mL DES Temperature: 40–60 °C Time: 30–50 min. US intensity: 250–350 W Control: Extraction with 80 % methanol for UAE	-The optimized yield was obtained when extracted for 41 min at 51 °C with a DES (15 % water v/v)of L-lactic acid and glycol in a 1:2 M ratio andan SSR ratio of 25 mL/g and ultrasonic power of 304 W. -UAE + DES yielded TPC of 7.38 mg FAE/g d.m. (67 % higher than UAE) and TFC of 4.30 mg RE/g d.m. (63 % higher than UAE).	[22]
UAE + DES	Foxtail millet bran	DES: Choline chloride/L-proline/Glycine/Betaine/ Glycerol + Oxalic acid/Lactic acid/Glycerol/L-lactic acid/Citric acid, Sodium acetate molar ratio: 1: 1, 1: 2, 1: 3, 2: 5, 1:1, 1:5, 3: 1 Water in DES(v/v): 20–40 % SSR: 1:10 ratio Temperature: 50–70 °C Time: 20–40 min US intensity: 200–300 W	-The highest yield was observed at betaine/glycerol in a 1:2 M DESs containing 29 %(v/v) water, 247 W, 61 °C for 31 min. -Corresponding yields of TPC was 7.8 mg FAE/g d.m. (2.25 times CSE), and TFC of 8.89 mg RE/g d.m. (2 times CSE).	[11]

US, ultrasound; UAE, ultrasound-assisted extraction; DES, deep eutectic solvent; HHP, High hydrostatic pressure; SSR, solid-to-solvent ratio; TPC, total phenolic content; TFC, total flavonoid content; GAE, grinding-assisted extraction; WBE, water bath extraction.

use of switchable hydrophilicity solvents (SHSs) with their ability to recover analytes effectively by switching between a hydrophilic (dissociated form) and a hydrophobic state (molecular form). This switch can be achieved through pH or temperature adjustments and other methods, permitting enhanced yield of target components [156]. The application of Deep Eutectic Solvents (DES) in millet bioactive compound extraction is a nascent but promising field. Studies demonstrate its efficacy in lignin extraction and pretreatment, yielding lower molecular weight phenolic compounds. Combinations of DES with antioxidants and extraction techniques significantly enhance the yield of compounds like β-carotene. High Hydrostatic Pressure (HHP) pretreatment coupled with DES extraction showcases a substantial increase in polyphenol yields. The use of Switchable Hydrophilicity Solvents (SHS's) adds versatility, offering a dynamic approach to recovering analytes efficiently. Future implications involve further exploration of DES applications, optimization of combined techniques, and the development of sustainable extraction protocols for millet bioactives.

6.3.4. Comparison between DES and ultrasound-assisted extraction techniques

The foxtail millet husk (FMH) had a notably higher level (5–17 times) of phenolics compared to the dehulled millet used for cooking [140]. Chunqing Wang et al. [22] employed DES with the UAE to extract phenols from foxtail millet husk, along with flavonoids, α-glucosidase, and acetylcholinesterase inhibitors. To extract a significant quantity of bioactive compounds, the UAE and DES were combined [22,157]. A significantly higher TPC (67.7 %), TFC (63.49 %), antioxidant activity, α-glucosidase, and acetylcholinesterase inhibitory activities (IC_{50}) was obtained for combined UAE and DES extracted mass compared to UAE alone. When UAE is integrated with DES, ultrasonication significantly disrupts the cell architecture of the fox millet husk, creating pores and cracks on sample surfaces as per the scanning electron microscopy (SEM) observations. This indicated significantly improved yield results for such combination

techniques. Further studies are required to assess the potential toxicity of the extract before its use in food applications [157]. Finger millet bran is composed of polyphenols, γ-oryzanol, phytosterols, and unsaturated fatty acids that exhibit greater antioxidant activity compared to millet grain [140,158]. To optimize the yield, a study explored ultrasonic-assisted deep eutectic solvent extraction of finger millet bran, utilizing various combinations of DES, ultrasonic intensities, and temperatures [20]. The yields of TPC and TFC were at least 200 % higher than the CSE method. Thus, ultrasonic-assisted DES extraction may offer a promising approach for enhancing the yield and bioactivity of finger millet bran extracts.

Although DESs have been in use for about two decades and have shown successful applications in various extraction processes, there is still a need for a deeper understanding of the underlying mechanisms involved. It is crucial to explore the interactions between DES and the target compounds and develop strategies to improve the transfer rates of the target compounds to the DES phase. Furthermore, comparative studies, comprehensive evaluations of efficiency, selectivity, and environmental impact of DES with traditional solvents are the future scope of research. The combination of DES with UAE proves effective in extracting bioactive compounds from foxtail millet husk, resulting in significantly higher yields and enhanced bioactivity. The SEM observations confirming structural disruption further validate the efficacy of the combined technique. Exploration of potential toxicity and optimization for food applications is crucial. Additionally, while DES applications exhibit success, a deeper understanding of underlying mechanisms, interactions, and comparative studies with traditional solvents is essential for advancing the field and ensuring sustainable and efficient extraction processes.

DES extraction techniques for bioactive compounds from millets present both advantages and challenges that require careful consideration. One of the primary advantages of DESs is their cost-effectiveness compared to ionic liquids, making them a more accessible option for research and industrial applications. Additionally, DESs are environmentally friendly as they are biodegradable and non-toxic, aligning well with the growing emphasis on green extraction technologies. Their safety, recognized by the FDA, allows for direct integration of DES extracts into food products, enhancing their nutritional value. Furthermore, DESs offer customizability, allowing researchers to tailor solvent properties to match the solubility or polarity requirements of specific target compounds, thus improving extraction efficiency and selectivity. These advantages make DESs a promising option for various industries, including food, pharmaceuticals, and nutraceuticals. However, some notable drawbacks and precautions merit attention. Despite their potential, the application of DES in extracting bioactive compounds from millets is still relatively new, necessitating further research to fully understand its efficacy and limitations. Toxicity concerns, although DESs are generally regarded as safe, require comprehensive assessment before widespread use in food applications to ensure consumer safety. Mechanistic understanding of DES extraction mechanisms is also essential for optimizing protocols and addressing any unforeseen challenges. Comparative studies comparing DES efficiency, selectivity, and environmental impact against traditional solvents are crucial for informed decision-making. To maximize the benefits of DES extraction while mitigating risks, studies should focus on thorough toxicity assessments, mechanistic studies, and continuous optimization of extraction protocols. This approach will ensure that DESs can be safely and effectively utilized in extracting valuable bioactive compounds from millets for various applications, contributing to sustainable and innovative practices in the food and pharmaceutical industries.

6.4. Supercritical fluid extraction

6.4.1. Principle and mechanism of extraction

When a fluid exceeds its critical point in temperature (T_c) and pressure (P_c), it becomes a supercritical fluid, a dense fluid with unique characteristics between a gas and a liquid. Its density mimics a liquid's, while its viscosity and diffusivity are akin to a gas's [158]. As a result, a supercritical fluid can function as a solvent similar to a liquid but with improved kinetics for mass transfer. Supercritical fluid extraction (SFE) can use a variety of solvents, including carbon dioxide, nitrous oxide, ethane, propane, n-pentane,

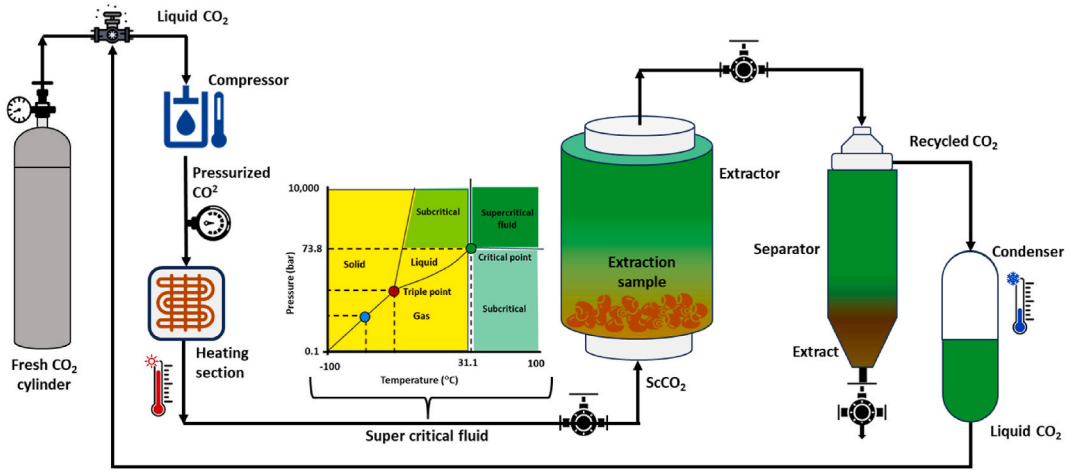


Fig. 6. A schematic showing the set-up and principle of supercritical fluid extraction of bioactive compounds from millet.

ammonia, fluoroform, sulfur hexafluoride, and water [159]. However, CO_2's critical point at 31.06 °C and 7.38 MPa is optimal for supercritical fluid extraction. It is the preferred solvent due to its favorable properties, such as being non-flammable, non-corrosive, cost-effective, and generally recognized as safe [160]. SFE is a method of extracting the selected component, called the extractant, from another component, called the matrix, using supercritical fluids as the extracting solvent. This extraction process typically occurs from a solid matrix but can also involve liquids [161]. The SFE process typically involves two primary stages: (1) extracting soluble components from solid substrates using a supercritical solvent and (2) separating the extracted compounds from the solvent after expansion [161]. A typical supercritical CO_2 extraction unit comprises a CO_2 pump, a sample holder, a pressure regulator, and a collecting vessel (Fig. 6). The solvent is heated to supercritical conditions. This supercritical CO_2 can diffuse into the sample, effectively solubilizing its constituents. The dissolved material is subsequently carried away from the extraction chamber into a separator operating at lower pressure, allowing the extracted substances to settle out. The CO_2 is then cooled down, recompressed, and recycled or released into the atmosphere [162].

6.4.2. *Supercritical fluid extraction of bioactive compounds from millets*

Supercritical fluid extraction (SFE) has become a popular extraction technique in the oil industry due to its ability to extract oils at low temperatures with high efficiency [163]. Table 5 represents a consolidated summary of studies using supercritical fluid-assisted techniques for the extraction of bioactive compounds from millets. Pang et al. [24] optimized the protocols for supercritical CO_2 extraction of oils from FMB while varying pressure (25, 30, 35 MPa), temperature (40, 45, 50 °C), and time (1.5, 2, 2.5 h). Higher sterols were observed compared to conventional solvent extract (CSE). The highest sterol found was β-sitosterol, comprising 56.2 % of the total sterols. Campesterol and stigmastanol were the next most abundant, each making up approximately 15.9 % and 15.1 %, respectively. Other phytosterols (or phytostanols), including ergostanol, stigmasterol, and fucosterol, were also present. Similarly, sorghum bran accounts for approximately 7 % of the total grain and is rich in non-starch polysaccharides, phenolic compounds, and natural wax, making it a valuable source of various compounds [164]. Black pericarp sorghum contains the highest levels of 3-deoxyanthocyanidins, which are highly resistant to oxidation compared to other anthocyanidins [165]. The black sorghum bran was subjected to supercritical CO_2 extraction, followed by a subsequent extraction using a mixture of 50 % (v/v) ethanol and water. SFE yielded the highest phenol yields (13.9 mg GAE/g bran) [25]. The SFE extract revealed the presence of four primary phenolic acids: cinnamic acid, caffeic acid, p-coumaric acid, and ferulic acid. Among the samples, the prevailing flavonoids were apigenin,

Table 5
Summary of studies using supercritical fluid-assisted techniques for the extraction of bioactive compounds from millets.

Method	Matrix	Process parameters	Inference	Reference
SCE	Foxtail millet bran	Pressure: 25–35 MPa Temperature: 40–50 °C Time: 1.5–2.5 h CSE with petroleum ether for 24 h at 60 °C with SSR of 1:10	-Optimal recovery (7.97 %) was obtained for 2.3h at 30.03 MPa, 47.93 °C. -The total sterols content of the oil was 1.55 % (1.4 times higher thanthe CSE yield)	[24]
SCE vs SPE	Foxtail millet bran	SCE: Pressure: 28 MPa Temperature: 40 °C Time: 2.5 h SPE: Pressure: 0.5 MPa Temperature: 40 °C Time: 1.5 h CSE with n-hexane for 3 h	Yields were 17.1 % (75 % of total oil) for CSE, 19.6 % (86 % of total oil) for CSE, and 21.7 % (96 % of total oil) for SPE.	[80]
SCE + ethanol extraction	Milled black sorghum bran	Pressure: 30 and 40 MPa Temperature: 40 and 60 °C Time: 3 h Followed by extraction with 100 % or 50 % (v/v) ethanol in water at 15 % (w/w) SSR	-The highest lipid yield of 5.7 % was obtained at 60 °C and 40 MPa. -SCE (3 h at 40 MPa and 40 °C) + 1:1 ethanol-water extraction resulted in a yield of 13.9 mg GAE/g bran.	[25]
SCE	Sorghum (whole kernel, bran, and dried distillers' grains with soluble [DDGS])	Pressure: 30 and 40 MPa Temperature: 50 and 70 °C Time: 0.5–5 h	-SCE at 40 MPa and 70 °C produced a higher wax yield of 4.9 % from DDGS compared to the whole kernel (0.6 %) and bran (3.3 %) fractions. -Whole kernel extracts had the highest wax (89 %)in SCE, while DDGS and bran had 53.3 % and 26.8 % wax, respectively, after SCE at 40 MPa and 70 °C.	[26]
SCE vs RSE	Sorghum distillers' grains	SCE: Pressure: 20 and 27.5 MPa Temperature: 40–70 °C Time: 4 h RSE: Solvent: n-hexane Temperature: 45–69 °C Time: 6 h	The total yield of lipids obtained after SCE at 27.5 MPa and 70 °C was 15 % (w/w), which is about 77 % higher than the RSE yield.	[81]

CSE, conventional solvent extraction; RSE, recirculated solvent extraction; SCE, supercritical CO_2 extraction; SPE, subcritical propane extraction; SSR, solid-to-solvent ratio.

apigeninidin, luteolinidin, 7-methoxy apigeninidin, and luteolin. This increased yield may be attributed to the solvency of SC-CO_2, which can be further optimized by incorporating GRAS co-solvents commonly used in phenolic compound extraction [166]. Sorghum also contains natural wax that could serve as a domestically grown substitute for Carnauba or a similar additive. Demand for natural waxes is increasing in the food packaging industry due to health concerns associated with paraffin wax [167]. A green supercritical CO_2 extraction was developed to extract natural wax from sorghum and its by-products, including sorghum dried distillers' grains with soluble (DDGS) and sorghum bran [26]. The impact of pressure on wax extraction from the whole kernel was greater at 70 °C than at 50 °C. There was no significant difference in the yield obtained at 30 MPa and 50 °C (0.5 %) and 40 MPa and 50 °C (0.4 %). However, the yield increased significantly to 0.8 % at 40 MPa/70 °C. The effect of temperature and pressure on yields was credited to the domination of the "vapor pressure effect" over the "solvent density effect" at a higher temperature and pressure combination [26].

Supercritical fluid extraction (SFE) emerges as a potent technique for extracting bioactive compounds from millets, offering high efficiency at low temperatures. Studies on foxtail millet bran and sorghum bran showcase the versatility of SFE in yielding sterols, phenols, and natural wax. The optimization of SFE parameters enhances the extraction of specific compounds, such as β-sitosterol and phenolic acids, providing valuable insights for the food and oil industries. The utilization of sorghum's natural wax as an alternative to conventional waxes presents an eco-friendly option for the packaging industry. As the demand for natural alternatives rises, SFE's role in extracting functional components gains significance. Future implications involve further optimization of SFE conditions, exploration of additional bioactive compounds, and the development of sustainable practices for diverse industrial applications. The potential of SFE in contributing to the sustainability and quality of millet-derived products is promising and warrants continued exploration.

6.4.3. Comparison of supercritical and subcritical fluid extraction

SCE and subcritical propane extraction (SPE) were compared using foxtail millet bran. Subcritical extraction is carried out below the critical point of a liquid. The pressure of SPE for oil extraction is lower by at least an order (one-tenth) of magnitude than supercritical extraction (SCE). SC-CO_2 extraction provided a higher yield (14.6 %) than solvent extraction; subcritical propane extraction (SPE) yielded significantly higher (11.6 %) amounts than SCE. This was attributed to the fact that subcritical propane is a superior solvent for triacylglycerols compared to supercritical carbon dioxide [23].

6.4.4. Comparison of supercritical and recirculated extraction

Sorghum-dried distillers' grains with soluble (DDGS) are the byproducts obtained during the fermentation of sorghum grains with *Saccharomyces cerevisiae* for ethanol production. With the valorization of this byproduct by extraction of lipids as a goal, a study [27] explored SC-CO_2 extraction and recirculated solvent extraction (RSE) at different temperatures and pressures. SC-CO_2 extraction yielded over 75 % higher than RSE. This difference might be attributed to the varying polar characteristics of the solvents. While hexane is a non-polar solvent, SC-CO_2 has intermediate polarity due to its large molecular quadrupole. Though it is a non-polar solvent, SC-CO_2 also has a limited affinity towards polar solutes. This enables it to dissolve more polar lipids, proteins, and sugars, resulting in higher extract yields than hexane [27,28].

SFE holds great promise as a technique that surpasses conventional methods. It offers several advantages, but research gaps still need exploration. One area of interest is finding alternative solvents and co-solvents to carbon dioxide (CO_2) that can enhance extraction efficiency and selectivity. By investigating other supercritical fluids, such as water, ethane, propane, or nitrogen, we can unlock new possibilities and expand the scope of this technique. Moreover, the combination of SFE with other extraction techniques holds the potential to improve overall efficiency. Future research should investigate the mechanisms and optimization of these combined techniques to further enhance their performance for commercial applications. SFE offers numerous advantages, making it a popular choice for extracting bioactive compounds from millets. Advantages of SFE include its high efficiency at low temperatures, allowing for the extraction of heat-sensitive compounds without degradation. CO_2 as a solvent is environmentally friendly and safe for food applications, aligning with green extraction principles. The versatility of SFE is evident in its ability to extract a wide range of compounds from millets, such as sterols, phenols, and natural waxes. These extracted components have various industrial applications, from functional food ingredients to eco-friendly packaging materials. The drawbacks and precautions that need to be considered include that SFE can be capital-intensive to set up initially, requiring specialized equipment and expertise. Optimization of SFE parameters is crucial for achieving desired extraction yields, which may require time and resources. There are also safety considerations when working with high-pressure systems, necessitating proper training and maintenance protocols to prevent accidents. Precautions in SFE involve ensuring the purity of the CO_2 solvent, as contaminants can affect the quality of extracted compounds. Proper handling of high-pressure equipment and adherence to safety guidelines are essential to prevent hazards. Additionally, thorough optimization of extraction conditions is needed to maximize yields and minimize energy consumption.

6.5. Enzyme-assisted extraction

6.5.1. Principle and mechanism of extraction

Enzymes are proteins made up of a single polypeptide chain; they act as biological catalysts and have a globular shape [168]. Enzymes can be obtained from different sources, such as bacteria, fungi, animal organs, or plant extracts and fruits. Intracellular enzymes are located inside the cell, while extracellular enzymes are released and can function outside the cell [169]. To extract bioactive materials from plants, it is often necessary to use a range of enzymes, including cellulases, pectinases, and hemicelluloses [170]. These enzymes work by breaking down the structural integrity of the plant cell wall, thus increasing the permeability of the cell wall components [171] (Fig. 7). By hydrolyzing the cell wall components, the enzymes enable a greater yield of bioactive materials to be extracted from the plant. The increased cell wall permeability ultimately results in a higher extraction yield of bioactive materials

[172]. Using this approach under mild process conditions can efficiently extract and release bioactive compounds from the cell wall matrix. Enzymatic reactions are typically carried out at lower temperatures ranging from 15 °C to 45 °C; this makes it a particularly useful tool in extracting compounds such as bioactive compounds that are heat sensitive [173]. Temperatures exceeding 60 °C can induce irreversible structural changes in enzymes because of the thermal effect on molecular vibrations. The vibrational effects can potentially modify the conformation of proteins and disrupt the internal cross-linkages in their polypeptide chains [174]. To ensure maximum enzyme activity, it is important to standardize the operational conditions for each application, including enzyme concentration, incubation time, incubation temperature, and optimal pH.

6.5.2. *Enzyme-assisted extraction of bioactive compounds from millets*

The enzyme-assisted extraction of bioactive compounds from millets involves the selection of enzymes that revolve around key principles. Firstly, enzymes must exhibit specificity, targeting the bonds relevant to the bioactive compounds of interest. For instance, proteases are chosen to break peptide bonds in proteins, while enzymes like amylases act on carbohydrate bonds [175]. Secondly, enzyme selection considers compatibility with optimal pH and temperature conditions for extraction, ensuring their activity and stability during the process [176]. Moreover, safety and regulatory compliance are crucial, with enzymes needing approval for food applications. Efficiency is also a priority, as enzymes should demonstrate high extraction yields of bioactive compounds from millets. Enzymes cleave specific bonds based on their type and function. For example, proteases cleave peptide bonds in proteins, producing peptides or amino acids [177]. The amylases break down α-1,4 glycosidic bonds in starch, while cellulases target β-1,4 glycosidic bonds in cellulose and lipases hydrolyze ester bonds in triglycerides, releasing fatty acids and glycerol [178]. By selecting enzymes that target these specific bonds in millet components, enzyme-assisted extraction efficiently releases bioactive compounds for various applications in the food and pharmaceutical industries.

A summary of studies using enzyme-assisted extraction of bioactive compounds from millets has been presented in Table 6. Bioactive peptides produce several biological effects at the tissue level by being absorbed through the intestine [179]. A study [40] reported that the protein hydrolysate from pearl millet bran showed a moderate decrease of 16 % in the DPPH assay results compared to BHT, indicating its potential antioxidant activity. Remarkably, the hydrolysate demonstrated over five times higher activity than the pearl millet protein isolate [40]. This difference in antioxidant activity can be attributed to bioactive substances within the pearl millet protein hydrolysate. Later, Agral et al. [43] utilized green tender sorghum as the substrate, employing alcalase (endo-protease) to fragment proteins. The hydrolysate was then fractionated using various techniques, such as ultrafiltration, gel filtration, and HPLC, to isolate the antioxidative peptides. The highest activities observed were for fractions labeled GF2 obtained by gel filtration. The antioxidant peptides (2–20 amino acids long) were formed because of the proteolysis of the parent protein [43]. The basis for these properties is the aromatic rings they carry, excessive donor electrons, and appropriate hydrophobic character.

Siddiq and Prakash [39] divided certain grain flour into coarse and fine fractions and measured the antioxidant capacity of dietary fiber, which comprises substances like polyphenols, flavonoids, and tannins. The authors examined the effect of enzyme treatment on finger millet, pearl millet, sorghum, and wheat. Finger millet exhibited a significant increase of 28–46 % in polyphenol levels while experiencing a 3 to 4-fold decrease in flavonoid levels and a 2.3 to 1.3-fold decrease in tannin levels. Similarly, pearl millet showed a 2–2.5-fold increase in polyphenols, accompanied by approximately 30 % and 17 % decrease in flavonoids and tannins, respectively, after enzyme treatment. Sorghum treated with enzymes displayed a remarkable over 5-fold increase in polyphenol content, with flavonoids increasing from undetectable to nearly 6 mg/100 g, and tannins increased by 2-fold. Wheat demonstrated approximately a 3-fold increase in polyphenols, a 2-fold increase in flavonoids, and a 2-fold increase in tannins following enzyme treatment. Overall, the antioxidant activity of the grains increased by 26 %–139 %. The enzyme treatment releases more active compounds from the grains, enhancing antioxidant activity [39].

Finger millet was subjected to combined enzyme treatment and ultrasonication to develop a sustainable approach to extract and utilize polyphenols in producing nutraceuticals and functional foods. The use of hydrolytic enzymes results in the breakdown of the cellulosic network, leading to improved distribution of solvents, reduced particle size, and enhanced mass transfer rate. These combined effects ultimately contribute to more efficient extraction of bound phenolics [180]. Balasubramaniam et al. [41] tried to improve and optimize the extraction of phenolic compounds from the seed coat of finger millet using enzyme treatments (xylanase and cellulase) and ultrasound assisted extraction. The process rate was three times higher, and the yields of polyphenols approximately doubled. The difference in results could be attributed to the solubility of flavonoids and tannins about the polarity of the solvent used

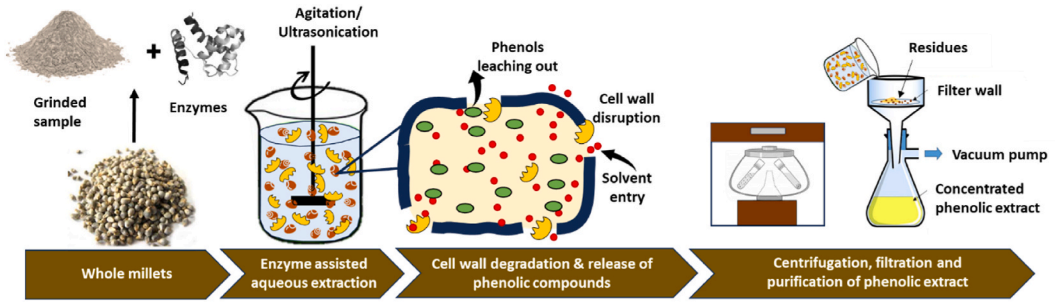


Fig. 7. A schematic showing the sequential steps for enzyme-assisted extraction of bioactive compounds from millet.

Table 6
Summary of studies using enzyme-assisted extraction of bioactive compounds from millet.

Method	Matrix	Process parameters	Inference	Reference
EAE	Pearl millet bran	Temperature: 37 °C Time: 3 h pH: 6.5 1 % trypsin	-The amino acid sequence of the peptide was SDRDLLGPNNQYLPK. -Protein from hydrolysate exhibited a higher radical scavenging capacity (78 % ABTS).	[40]
EAE	Green tender sorghum	Temperature: 50 °C Time: 5 h pH:9.5 1 % alcalase	-The antioxidant activity for protein hydrolysate was 61.7 % DPPH assay and 78.6 % ABTS. -The highest activity observed for GF2 was obtained by gel filtration.	[43]
EAE	Finger millet, Pearl millet, Sorghum, and Wheat	Temperature: 37 °C Time: 2 h pH:2.0 0.12 % pepsin + Pancreatin with pH 7 and incubated for2h	-Finger millet showed about 28 % increase in polyphenols and 1.3-fold drops in tannin levels after EAE. - Pearl millet showed a 2-fold increase in polyphenols and about 17 % drop in tannins after EAE. -Sorghum displayed a 5-fold increase of polyphenols while tannins increased by 2-fold after EAE. -Wheat polyphenols were increased by 3-fold, and tannins were elevated by 2-fold after EAE.	[39]
EP-UAE	Finger millet seed coat	Enzyme: Xylanase and cellulase SSR: 10%w/v Temperature: 50 °C Time: 2 h + Solvent: 50 % ethanol US power: 500 W Time: 30 min	-Polyphenol yields were increased by 1.8 and 2.2 folds by xylanase and cellulase pretreatment, respectively, while reducing the time by three-fold and solvent consumption.	[41]
EP-UAE vs. UAE	Proso millet	EP: 1 % α-amylase + protease treatment for 30 min UAE: Temperature: 50 °C Time: 7 min US power: 150W Frequency: 37 kHz	-The dietary fiber yield was increased from 38 % to 94 % by EP-UAE compared to UAE treatment.	[45]
EP-UAE	Finger millet	Enzyme: β-glucosidase Solid to enzyme extract ratio: 0.3:1 to 0.6:1 w/v Temperature: 30–60 °C Time: 1–4 h	Maximum polyphenol release was noticed at 2.327 mg/mL for 0.3:1 at 30 °C.	[42]
EP-UAE	Foxtail millet	Liquid-solid ratio: 25:1 to 35:1 Temperature: 55–65 °C Time: 10–30 min	The polysaccharide yield was 3.87 % with a liquid:solid ratio of 31:1 when hydrolyzed at 59 °C for 22 min.	[44]

EAE, enzyme-assisted extraction; EP-UAE, enzyme pretreated ultrasound-assisted extraction; UAE, ultrasound-assisted extraction; SSR, solid-to-solvent ratio.

and the potential thermal degradation of phenolic compounds during retort processing [121].

The primary dietary fiber components in millet include hemicellulose, lignin, cellulose, cutin, and silica [181]. Among various sources and raw materials, it was observed that the enzymatic extraction method assisted by ultrasound was the most effective. The extraction process involved treating the sample with 1 % α-amylase for a specific duration, followed by adding protease for an additional 30 min. Subsequently, ultrasonicated at 50 °C for 7 min. The yield percentages ranged from 18.5 % in whole-seed quinoa to 94.2 % in millet flour [45]. Ultrasonic extraction significantly increased the dietary fiber content in flour fractions by more than 2.4 times compared to the control method. Similarly, the dietary fiber content in whole seed fractions increased by 1.6 times. However, the ultrasonication process may cause damage to the cell walls, which could negatively impact the extraction yields of soluble dietary fiber (SDF), especially when present in high amounts [182]. Ultrasonication facilitates water penetration into the cell wall structure, thereby increasing the accessibility of constituents such as starch to enzymatic digestion in the subsequent extraction step [45]. The dietary fiber is liberated from the structures of starch and proteins during the extraction process. However, simple ultrasonication was not an effective method for extracting SDF in the examined raw materials, particularly in the case of millet samples [183].

Yadav et al. [42] explored the extraction and evaluation of the antioxidant properties of the total polyphenol content in samples derived from fermented and enzymatically treated fractions of finger millet. For the extraction of polyphenols, 80 % ethanol was employed on the dried fermented biomass of finger millet. The activity of β-glucosidase enzymes began to decline after the 6th day of incubation. The highest release of polyphenols was observed at concentrations of 2.32 mg/mL for a ratio of 0.3:1 and 2.23 mg/mL for a ratio of 0.6:1, both at 30 °C [42]. The β-glucosidase facilitated the release of phenolic compounds attached to the terminal ends of wall polysaccharides. As a result, the polyphenols are freed from their linkages with cell wall polymers, increasing the total polyphenol content. Zong et al. [44] found that the yield of millet bran polysaccharide extraction using enzymes was higher (3.87 %) compared to extraction without enzymes (about 1.8 %). The maximum yield of polysaccharide was achieved when the ratio of pectinase to cellulase was 1:4 (g/g). The cell wall of millet rice bran consists of 20 % pectin and 80 % cellulose. Pectinase acts on the intercellular space,

breaking down pectin macromolecules into smaller molecules to facilitate cell separation. In contrast, cellulase acts on the cell wall, breaking it down to release the polysaccharides. Complex enzyme mixtures generally exhibit higher efficiency compared to single enzymes. Therefore, the optimal complex enzyme ratio for extracting millet bran polysaccharide was 1:4 (g/g), resulting in a nearly threefold increase in polysaccharide yield under optimal conditions [44].

EAE shows great potential in improving the extraction of bioactive compounds from various sources. Developing customized protocols tailored to different substrates is essential, especially when dealing with complex matrices and challenging compounds. Additionally, exploring new sources and recovering valuable compounds from industrial by-products can greatly broaden the applications of EAE. Furthermore, proper pre-treatment of the substrate is crucial, and future studies could focus on developing sustainable and efficient methods like microwave-assisted pre-treatment and ultrasonication. By tackling these research gaps and limitations, EAE can achieve higher efficiency and effectiveness in extracting bioactive compounds from diverse sources. Enzyme-assisted extraction (EAE) proves to be a promising and the best technique for extracting bioactive compounds from millets, offering enhanced antioxidant activities and improved release of polyphenols. Various enzymes, such as alcalase, xylanase, cellulase, and β-glucosidase, contribute to the breakdown of complex structures, facilitating the extraction of valuable compounds. Studies on pearl millet, sorghum, finger millet, and other millet varieties demonstrate increased polyphenol levels, antioxidant activities, and dietary fiber content through EAE. The combination of enzymes with ultrasonication further amplifies the efficiency of polyphenol extraction. Future implications involve customizing EAE protocols for different substrates, exploring new sources, and addressing challenges in complex matrices. Sustainable pre-treatment methods, like microwave-assisted and ultrasonication, could enhance EAE's sustainability and efficiency. Overcoming these challenges can unlock the full potential of EAE in extracting bioactive compounds, contributing to diverse applications in the food and pharmaceutical industries.

Enzyme assisted extraction (EAE) presents several advantages in extracting bioactive compounds from millets. One key advantage is its specificity, as enzymes target specific bonds relevant to the bioactive compounds of interest, leading to higher extraction yields. Additionally, EAE operates at lower temperatures or negligible heating, preserving heat-sensitive compounds, and is environmentally friendly. The enzymatic breakdown of complex structures enhances the release of valuable compounds like polyphenols and antioxidants, contributing to improved nutritional profiles and functional properties of millet extracts. The drawbacks of the technique are related to enzyme selection must align with optimal pH and temperature conditions for extraction, and safety concerns regarding enzyme activity and regulatory compliance are paramount. Ensuring the purity and stability of enzymes, along with standardized operational conditions, is crucial for consistent and efficient extraction results. While EAE offers enhanced extraction efficiency, its effectiveness may vary depending on the substrate and enzyme combination used, requiring customized protocols for different millet varieties. Proper pre-treatment of substrates, such as ultrasonication, and addressing challenges in complex matrices are areas that merit further exploration to optimize EAE's sustainability and efficacy in extracting bioactive compounds from millets.

6.6. *Accelerated solvent extraction*

6.6.1. *Principle and mechanism of extraction*

Pressurized liquid extraction (PLE), also known as accelerated solvent extraction (ASE), pressurized solvent extraction (PSE), and pressurized fluid extraction (PFE), is an automated method used to extract analytes from solid and semi-solid samples [184]. It operates at temperatures higher than the boiling point of most solvents and uses pressure to keep the solvents in a liquid state

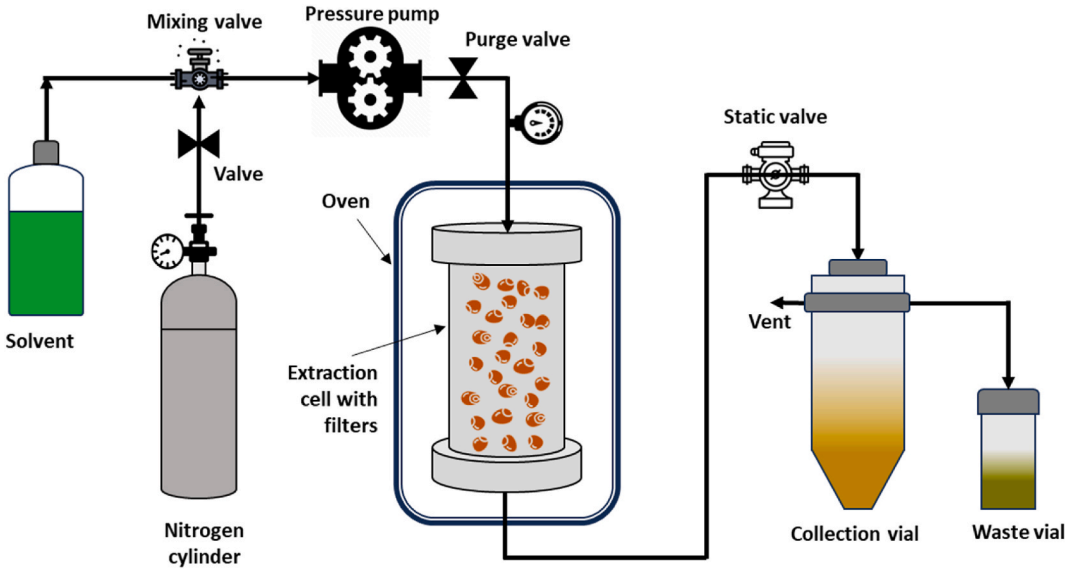


Fig. 8. A schematic showing the set-up for pressure solvent extraction of bioactive compounds from millet.

throughout the process. This technique enables efficient filling of the extraction cell, facilitates solvent penetration into sample pores, and maintains the solvent in liquid form at operating temperatures [185]. Various solvents like methanol, water, toluene, dichloromethane, ethyl acetate, and acetonitrile can be used in ASE for food. The selection of the extraction solvent should consider properties such as boiling point, polarity, specific density, and toxicity [186]. The process of extracting analytes from solid samples involves three key steps: desorption of analytes from solid particles, diffusion of analytes through the solvent within the particle pores, and transfer of analytes to the flowing fluid (Fig. 8). Extraction typically takes 15–25 min and uses a small amount of solvent. Higher temperatures accelerate extraction kinetics, while elevated pressure ensures efficient and safe extraction [187]. Dry, finely divided solid samples are preferred to facilitate the flow of the extraction solvent and ensure thorough permeation of the matrix particles. Grinding larger particle sizes (>1 mm) can increase the interaction surface between the solvent and the matrix, enhancing extraction efficiency. For wet or sticky samples, drying agents such as sodium sulfate, pelleted diatomaceous earth, or dispersing agents like Ottawa sand can be added before extraction [185].

There are two primary configurations for ASE: static and dynamic instruments. In the dynamic system, high-pressure pumps facilitate the continuous circulation of the extraction solvent through the sample vessel [188]. In this mode, the solvent is constantly pumped through the sample matrix extractor while the outlet valve remains open throughout the extraction process [188]. On the other hand, in the static set-up, the extraction process operates in a batch mode, involving one or more extraction cycles by introducing fresh solvent between each cycle [189]. Static mode pressurizes the extractor through the solvent inlet while keeping the outlet valve closed. Once the extraction is complete, the valve is opened, allowing the solvent-extract mixture to be released and collected [190].

6.6.2. Accelerated solvent extraction of bioactive compounds from millets

Higher pressures (>1000 psi) in accelerated solvent extraction (ASE) facilitate rapid heating of solvents beyond their boiling point, resulting in enhanced diffusion rates, disruption of solute matrix interactions, and reduced solvent viscosity, thus facilitating improved penetration into the matrix, and leading to improved extraction efficiency. The literature on applying ASE of bioactive compounds from millets is currently limited. Barros et al. [46] evaluated the extractability of sorghum phenolic compounds using ASE compared to CSE. The extraction at 150 °C using 50 % and 70 % ethanol/water (v/v) demonstrated notable efficacy in extracting the identical quantity of phenolic compounds (45 mg GAE/g) and marginally higher antioxidants (628 mmol TE/g) from black sorghum compared to CSE methods(560 mmol TE/g). ASE extraction at temperatures above 100 °C (120 °C and 150 °C) significantly increased the levels of extractable phenols and antioxidants in black sorghum extracts. This is because of increased diffusion rates, disruption of some of the solute matrix interactions, and formation of phenols during the Maillard reaction.

Table 7
Summary of studies exploring the accelerated solvent extraction of bioactive compounds from grains.

Method	Matrix	Process parameters	Inference	Reference
ASE	Black Sorghum	Pressure: 1500 psi Static time: 1 min Temperatures: 60–150 °C Solvents: 50–70 % v/v ethanol/ water or acidic water (pH 2.5)	Highest phenols (45 mg GAE/g) and antioxidant compounds (628 mmol TE/g) extracted at 150 °C using 50 % and 70 % ethanol/water (v/v)	[46]
ASE and MAE	Black rice	ASE Pressure: 1500 & 2500 psi Static time: 5 & 10 min Temperature: 25 & 50 °C MAE Power: 300 & 1200 W Temperature: 50 & 70 °C Time: 10 & 20 min	-ASE at 50 °C, 2500 psi, for 10 min using 5 cycles and a 100 % flush volume yielded 3379 μg/g of anthocyanins. -MAE at 50 °C, 1200 W, and 20 min extracted 3485 μg/g of anthocyanins.	[135]
ASE	Rice	Pressure: 100–200 atm Temperature: 100–200 °C Static time: 5–10 min Flushing: 50–100 % Purge time: 60–120 s Sample: 2.5–5.0 g Solvent: 80–100 % ethyl acetate in methanol	-All the phenolics were extracted for 10 min with 2.5 g samples in 60 % ethyl acetate in methanol at 190 °C, 200 atm, and 100 % flush volume	[186]
ASE, UAE, and CSE	Rice berry bran (RBB)	ASE: Pressure: 100–200 atm Temperature: 60–80 °C Static time: 10–15 min Sample: diatomaceous earth in a 1:1 ratio UAE and CSE: Temperature: 60–80 °C Sample to ethanol: 1:20 w/v Time: 15–45 min	-ASE offered TPC and TAC of 19.70 GAE/g and 885.4 μg/g, respectively. -ASE reduced solvent requirement by 90 %, extraction time by 3 times, and increased extraction of TPC by 3 times and TAC by 4 times compared to UAE and CSE.	[187]

ASE, accelerated solvent extraction; CSE, conventional solvent extraction; MAE, microwave-assisted extraction; SSR, solid-to-solvent ratio; TAC, total anthocyanin content; TPC, total phenolic content; UAE, ultrasound-assisted extraction; US, ultrasound.

Substantial research has been conducted on ASE in rice, and interesting findings have been summarized in Table 7. ASE in rice appears to be an interesting approach to extracting phenolic compounds due to several benefits associated with high pressure and temperature conditions. Setyaningsih et al. [191] demonstrated that 100 % extraction of phenolics from rice was achieved for ASE with a solvent mixture of 60 % ethyl acetate in methanol at 190 °C and 200 atm. In another study on ASE in black rice, under optimal conditions (10 min extraction, with 5 cycles and 100 % flush volume at 50 °C and 2500 psi), successful extraction of anthocyanin pigments (3379 μg/g) was achieved. However, the results were slightly lesser than MAE (3485 μg/g) [136]. This difference can be attributed to the distinct chemical structures of the phenolic compounds and their varying reactivities in terms of stability when subjected to higher extraction temperatures. A comparative study between ASE, UAE, and CSE on extracting phenolic acids and anthocyanins from rice berry bran (RBB) was performed [192]. The ASE method offered several benefits, such as a reduction in solvent consumption by up to 90 %, a 3-fold reduction in extraction time, a 3-fold higher extraction of TPC, and a 4-fold high extraction of TAC compared to UAE and CSE. In addition, the yield of phenolics and anthocyanins was 3–5 times higher in ASE compared to UAE and CSE. Concomitantly, the prolonged high-temperature extraction is not ideal due to the hydrolysis of glycoside compounds and the formation of unstable aglycones or due to the hydrolytic opening of the heterocyclic ring that would form chalcone in the case of anthocyanins [143].

These studies affirm the potential of ASE as a sustainable extraction technique. Consequently, a burgeoning interest in ASE has been driven by increasing environmental concerns and demand for greener extraction practices. However, scaling up ASE from laboratory to industrial levels may present challenges, necessitating research to assess scalability and implement necessary modifications. Furthermore, the synergistic effects of combining ASE with other extraction techniques, such as ultrasound or microwave-assisted extraction, can be investigated to enhance efficiency, yield, and rapid extraction. The above studies demonstrate the ASE technique's efficacy in extracting phenolic compounds from sorghum and rice, showcasing benefits such as increased diffusion rates and disruption of solute matrix interactions. While limited literature exists on ASE in millets, studies on rice suggest its potential for achieving high extraction yields and reducing solvent consumption. The environmental sustainability of ASE aligns with growing concerns, but scaling up from laboratory to industrial levels poses challenges requiring further investigation. Future implications involve assessing scalability, exploring synergistic effects with other techniques, and optimizing ASE protocols for diverse millet varieties. Addressing these aspects will unlock the full potential of ASE, contributing to greener and more efficient bioactive compound extraction practices.

This method offers several advantages such as its ability to operate at higher pressures and temperatures accelerating the extraction kinetics, leading to enhanced diffusion rates and improved extraction efficiency. ASE also reduces solvent consumption and extraction time compared to conventional methods, making it a more environmentally sustainable option. However, the elevated temperatures in ASE can lead to the hydrolysis of glycoside compounds and the formation of unstable compounds, impacting the stability of extracted bioactive compounds. Additionally, scaling up ASE from laboratory to industrial levels may pose challenges that require careful assessment and modifications. Ensuring proper calibration of extraction parameters, such as pressure, temperature, and solvent composition, is crucial to obtaining consistent and reliable results. Furthermore, integration of Accelerated Solvent Extraction (ASE) with ultrasound or microwave-assisted extraction (MAE) can create synergistic effects that enhance extraction efficiency, yield, and speed. Ultrasound and MAE can disrupt cell structures, increasing solvent penetration and extraction rates during ASE. This combination optimizes the extraction process, leading to higher yields of bioactive compounds in a shorter time frame. Investigating these synergies allows for the development of more effective and rapid extraction methods, benefiting industries reliant on efficient extraction of valuable compounds from natural sources like millets. The studies on comparison and/or integration of ASE with UAE or MAE are limited in the literature. However, the proposed prospect might suggest developing a hybrid approach to leverage their synergistic effects and optimize extraction outcomes for bioactive compounds in millets.

6.7. Pulsed electric field

6.7.1. Principle and mechanism of extraction

The pulsed electric field (PEF) technique is a non-thermal method that employs short-duration pulses with high electric fields, typically ranging from 10 to 80 kV/cm and lasting from microseconds to milliseconds. The number of pulses applied to the product determines the effectiveness of the process [193]. The samples are placed between two electrodes and subjected to a high-voltage electric field. The specific parameters for PEF vary depending on the desired process, including electric field intensity (kV/cm) and specific energy (J/kg) [194]. PEF can be applied using different methods, such as exponentially decaying waves, bipolar waves, and oscillatory pulses, and implemented at different temperature conditions. PEF causes transformation or rupture of the cell membrane, increasing its electrical conductivity and permeability, thus resulting in reversible electroporation and enhancing extraction [195]. Effective extraction involves low specific energy (1–10 kJ/kg) and pulse cycles of nanoseconds to milliseconds [196]. Delicate plant tissues require electric field strengths (0.1–10 kV/cm), while tougher materials like seeds need higher intensities (10–20 kV/cm) for efficient extraction. The magnitude of the electric field strength directly impacts the extent of damage caused [196].

A PEF unit consists of a high-voltage pulse generator, a treatment chamber with a fluid monitoring assembly, and a monitoring/control system (Fig. 9). The generator includes a charger for converting AC to DC and an energy storage device. The switch in the generator activates and deactivates the high-voltage circuit to generate electric pulses. The treatment chamber has two electrodes separated by insulating materials, with the sample placed in the gap. The effectiveness of the treatment depends on electrode distance, applied voltage, and chamber geometry, which determine the electric field strength and energy delivery [197]. PEF treatment parameters for extraction can be categorized into high ($E > 1$ kV/cm), medium ($E \sim 0.1$–1 kV/cm), and low ($E < 0.1$ kV/cm) electric fields [198]. PEF extraction can be performed in batch [199] or continuous systems [196].

6.7.2. Pulsed electric field (PEF)-assisted extraction of bioactive compounds from millets

Lohani and Muthukumarappan [200] assessed the impact of flour-to-water ratio, electrical field intensity, and treatment time on the extraction of total phenolic content (TPC) and antioxidant activity (AA) of sorghum flour. Increasing the solid concentration from 10 % to 45 % (w/v) increased the electrical conductivity from 770 μS/cm to 1560 μS/cm. The higher electrical conductivity promoted electroporation and release of phenolics into the sample, influencing the TPC and AA [201]. Increasing the electric field intensity from 2 kV/cm to 3 kV/cm had no significant effect on TPC and AA. Despite the higher average specific energy of 8.99 kJ/kg transmitted during the PEF treatment at 3 kV/cm, the energy efficiency was reduced due to increased tissue disintegration caused by the higher electrical conductivity (2023 μS/cm). As a result, there was no further release of phenolic compounds. The higher energy input (10.8 kJ/kg) at 3 kV/cm may have caused lethal damage to the cells, resulting in irreversible loss of cell membrane permeability and subsequent degradation of the phenolic compounds [202]. The optimal condition was the extraction with a flour-to-water ratio of 45 % (w/v), an electric field intensity of 2 kV/cm, and a treatment time of 875 μs. Under the optimal conditions, the TPC increased by 24.8 %, and the AA increased by 33.9 % compared to the control sample. Furthermore, an increase in treatment time from 500 μs to 875 μs resulted in a significant increase of 5.7 % and 6.3 % in TPC and AA, respectively, for the sorghum flour. The number of pulses applied to the sample increased with longer treatment times. At 875 μs, the number of pulses induced a metabolic response by opening pores in the cell membrane, resulting in the release and absorption of phenolic compounds. However, further increasing the number of pulses to 1250 μs treatment time could irreversibly break the cell wall and damage the membrane, preventing further release of phenolic compounds [203].

The literature using PEF to extract bioactive compounds from millets is currently limited. Nevertheless, studies are exploring PEF-assisted extraction of bioactive compounds from rice, as summarized in Table 8. Salee et al. [204] compared the effects of pulse electric field (PEF), microwave, and ultrasonic-assisted water extractions on bioactive compounds in black rice. PEF with 3000 pulses and microwave treatment yielded high concentrations of TPC (486.8 and 486.2 mg GAE/g, respectively). The extract from PEF with 3000 pulses exhibited significantly higher DPPH scavenging activity and sirtuin1 enzyme stimulating activity than microwave and ultrasonic extractions. Another study by Salee et al. [205] optimized PEF treatment parameters such as the intensity of the electric field, the number of pulses, and the solid/water ratio to enhance the extraction yield of cyanidin-3-glucoside (C3G) and peonidin-3-glucoside (P3G) from black rice grain using PEF. The optimized conditions (5 kV/cm, 3000 pulses, 0.5 g/mL) resulted in higher yields of C3G (92.59 mg/g) and P3G (4.59 mg/g) compared to the untreated sample. Additionally, PEF extract exhibited the ability to stimulate sirtuin1 enzyme activity.

Similarly, Rajchasom et al. [206] applied PEF to extract anthocyanins from purple rice. They observed that the total antioxidant capacity (TAC) also increased as the number of pulses increased. The TAC peaked under both low (300 pulses at 4 kV cm^{-1} at 1.17 mg/L) and high (5000 pulses at 6 kV cm^{-1} at 3.28 mg/L) PEF treatments. Another study was conducted to extract phenolic compounds, flavonoids, and anthocyanins from brown rice using electric field intensity (1–3 kV/cm), pulses (10–1000), pulse duration (100 μs), and pulse repetition frequency (5 Hz) [207]. At 2 kV/cm and 1000 pulses, there was a 50 % increase in antioxidant activity. To summarize, pulsed Electric Field studies indicated the impact on total phenolic content (TPC) and antioxidant activity (AA). Optimizing PEF parameters, including flour-to-water ratio, electric field intensity, and treatment time, showcased increased TPC and AA in sorghum flour. The research on PEF-assisted extraction from millets is currently limited, but promising findings from studies on rice highlight its efficacy in enhancing TPC, DPPH scavenging activity, and enzyme-stimulating activity. PEF exhibits potential in extracting anthocyanins, phenolic compounds, and flavonoids from various rice varieties. Future implications involve extensive exploration of PEF on different millet varieties, optimizing parameters for maximum bioactive compound extraction, and tailoring strategies for specific applications. PEF's ability to induce a metabolic response, open cell pores, and enhance extraction efficiency

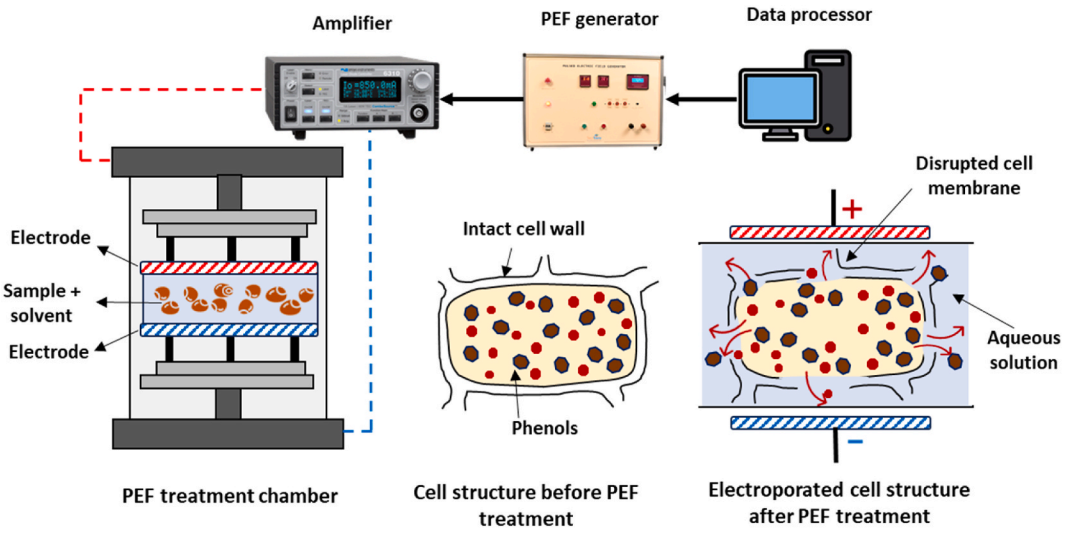


Fig. 9. A schematic showing the set-up and principle of pulse electric field (PEF) assisted extraction of bioactive compounds from millet.

Table 8
Pulsed electric field (PEF) assisted extraction of bioactive compounds from grains.

Matrix	Process parameters	Inference	Reference
Purple rice	Pulses: 0-500 Intensity: 2–4 kV/cm Frequency: 1 Hz	Poor anthocyanin content (0.7 mg/L) obtained at 4 kV and 100 pulses	[201]
Purple rice	Pulses: 1000-5000 Intensity: 6 kV/cm Frequency: 1 Hz	-Optimal anthocyanin content (3.28 mg/L) was obtained at 6 kV and 3000 pulses	[201]
Black rice	Pulses: 1000-3000 Intensity: 3–5 kV/cm Biomass-to-waterratio: 0.1–0.5 g/mL	Higher levels of cyanidin-3-glucoside (92.59 mg/g) and peonidin-3-glucoside (4.59 mg/g) were obtained at 5 kV/cm with 3000 pulses a biomass to water ratio of 0.5 g/mL	[200]
Black rice	Pulses: 1000-3000 Intensity: 5 kV/cm	-PEF at 5 kV/cm and 3000 pulses and microwave treatments (800 W/20 min) yielded high concentrations of total phenolic compounds and cyanidin-3-glucoside. -Antioxidant activity in the extract was 30.8, 28.3, and 25.3 % after PEF (3000 pulses/5 kV/cm), microwave (800 W/20 min), and ultrasound (60 °C/20 min) treatments, respectively.	[199]
Brown rice	Pulses: 10-1000 Intensity: 1–3 kV/cm Pulse width: 100 μs Frequency: 5 Hz.	-PEF of 2 kV/cm and 1000 pulses showed highest antioxidant activity (50 %). -Phenolic compounds detected were protocatechuic acid, α-tocopherol, 4-hydroxybenzoic acid, γ-oryzanol, γ-oryzanol, chlorogenic acid, vanillic acid, caffeic acid, syringic acid, cyanidin-3-galactoside, cyanidin-3-glucoside, cyanidin-3-rutinoside, *p*-coumaric acid, ferulic acid	[202]

underscores its role in advancing millet-based bioactive compound extraction.

The PEF extraction offers notable advantages as it operates as a non-thermal method, minimizing heat-related degradation of sensitive compounds. PEF can enhance extraction efficiency by promoting reversible electroporation of cell membranes, leading to increased permeability and release of bioactive from cells. This technique allows for precise control over parameters like electric field intensity, treatment time, and pulse cycles, optimizing extraction conditions for specific compounds. Furthermore, PEF can be integrated into batch or continuous systems, offering flexibility in processing. However, prolonged or excessive PEF treatment may cause irreversible cell damage, impacting the quality and stability of extracted compounds. Optimal parameters must be carefully determined to avoid over-processing. Additionally, equipment complexity and energy requirements can be significant, necessitating proper calibration, training, and maintenance. Ensuring uniform treatment across samples and standardizing protocols are crucial for reproducibility and consistency in results. Furthermore, studies on PEF-assisted extraction from millets are relatively limited compared to other techniques, highlighting the need for further research to fully understand its efficacy, scalability, and potential impacts on bioactive compound integrity.

6.8. Other pretreatments and assisted extraction techniques

6.8.1. Irradiation-assisted extraction

Food irradiation refers to exposing food to ionizing radiation [208]. Linear accelerators or Van de Graaff generators are utilized to accelerate electrons, and when these accelerated electrons interact with a heavy metal film, they generate X-rays [209]. X-rays have notable energy levels, up to a maximum of 5 MeV, allowing them to easily penetrate the cellular structure of plants [210]. Electron beam irradiation utilizes a stream of high-energy electrons to irradiate the sample. Similarly, gamma rays emitted by radioisotopes like ^{60}Co or ^{137}Cs also possess strong penetration capabilities [211]. The radiation after penetrating materials can effectively disrupt the cellular structures, promote the decomposition of chemical bonds, and result in the release of targeted compounds. Gamma irradiation can cause structural changes in plant cell walls, leading to increased permeability and easier release of targeted compounds during extraction. Irradiation as a pretreatment was applied to enhance the extraction of polyphenols, flavonoids, and other beneficial compounds from various grains, such as sorghum [212]. The treatments with gamma radiation doses at 1 kGy and 2 kGy had a positive effect on TPC proportional to the dose. The enhancement in the extraction of phenolic compounds results from the hydrolysis of phenolic glycosides and the subsequent liberation of aglycone [213]. The radiation-based extraction techniques offer an improved extraction, reduced extraction time, and preservation of heat-sensitive compounds. However, it is important to optimize the irradiation dosage, duration, and wavelength to ensure extraction efficiency and maintain the quality of the bioactive compounds.

Irradiation-assisted extraction presents several advantages in the extraction of bioactive compounds from food materials. It can effectively disrupt cellular structures, enhancing the release of targeted compounds such as polyphenols and flavonoids. This method often leads to improved extraction efficiency, reduced extraction time, and preservation of heat-sensitive compounds, making it suitable for delicate bio-actives. Irradiation can penetrate plant cell walls, increasing their permeability and facilitating the extraction process. Additionally, irradiation can induce structural changes that aid in the liberation of compounds of interest. However, optimizing irradiation dosage, duration, and wavelength is crucial to ensure a balance between extraction efficiency and avoiding overdosage treatment that may lead to the degradation of bioactive compounds, which is yet to be explored. The studies related to the application of irradiation in millet extraction are limited. Regulatory compliance and safety standards regarding food irradiation must be strictly followed to ensure the quality and safety of extracted compounds. Furthermore, irradiation setup is expensive, and processes require specialized handling and expertise, adding to operational complexities. Careful monitoring and control of irradiation parameters are necessary to maintain the integrity and bioactivity of extracted compounds, highlighting the importance of rigorous quality control measures in irradiation-assisted extraction processes.

6.8.2. Mechanochemically assisted extraction

Mechanochemically assisted extraction (MCAE), an advanced pre-extraction method, is a technique that utilizes high-speed grinding to pulverize plant materials, including their tissues and cell membranes. This process plays a crucial role in effectively releasing intracellular molecules. These molecules can then interact with chemical auxiliaries, leading to the formation of chemical bonds. Consequently, this interaction facilitates the extraction of target compounds, resulting in improved extraction efficiency and enhanced hydrophilicity [214]. For instance, calcium phytate was extracted from rice bran enzymatically with the aid of MCAE; the yield was over 3-fold, with a 17-fold reduction in processing time than enzymatic treatment alone [215]. The MCAE technology comprises three main steps: (1) initial mechanical preparation of the raw material (sample preparation); (2) mechanochemical treatment of pre-activated materials using a solid reagent, wherein they are ground under high mechanical pressure in a ball mill; (3) subsequent extraction procedures. Through this mechanochemical treatment, the raw material undergoes both physical and chemical transformations, leading to an enhanced water solubility of the target compound. Due to its high efficiency, low operating temperature, and reduced or negligible consumption of organic solvents, MCAE has garnered significant attention in the food and pharmaceutical industries [216]. The potential mechanisms underlying the MCAE technique include reducing particle size, breaking down the cell wall, decomposing cellulose, and accelerating the dissolution kinetics. These mechanisms collectively contribute to increasing the extraction efficiency and reducing the processing time [217].

7. Comparison of extraction techniques and recommendations

The various extraction techniques explored for millet bioactive compounds offer a range of advantages and considerations, presenting a diverse knowledge for the selection of extraction methodology (Fig. 2). Among several techniques explored for millet bioactive compounds, UAE and MAE stand out for their rapid extraction capabilities, yielding high quantities of compounds with good purity. However, the energy consumption in UAE and MAE processes tends to be higher compared to other methods. DES technique exhibits promises with its eco-friendly nature, offering moderate yields and purity, although requiring a deeper understanding of mass transfer rates and mechanisms for broader applications. SFE showcases potential in preserving compound quality but requires further assessment of stability and shelf life for commercial viability. The PEF-assisted technique presents a non-thermal approach with promising results, although necessitating further optimization of parameters for millet-specific applications. PEF and MAE demand relatively higher energy for producing high-voltage pulses and microwave radiation, respectively. In addition, conventional methods such as Soxhlet and traditional solvent extraction techniques also consume significantly higher energy due to their prolonged heating and solvent evaporation processes.

Enzyme assisted extraction (EAE) displays considerable potential, especially for challenging compounds (polyphenols and anthocyanins), leveraging enzymes to enhance extraction efficiency and yield. EAE technique has the lowest energy input compared to any other technique. This is because of milder operating conditions and enzymatic reactions that catalyze the breakdown of cell walls and complex molecules, without the need for high temperatures or external force. Mechanochemically assisted extraction (MCAE) demonstrates high efficiency and reduced processing time by utilizing high-speed grinding to release intracellular compounds. This method, with its capacity to significantly reduce processing duration, presents an appealing option in terms of time efficiency. Despite each technique involving distinctive benefits, an ideal technique often involves a combination of these techniques considering specific compound characteristics, extraction efficiency, purity requirements, and operational costs. The integration of techniques and their optimization may provide synergistic effects, enhancing extraction efficiency and addressing the unique demands of extracting bioactive compounds from millet. Considering the energy efficiency, the enzyme assisted extraction technique may be regarded as a greener and more energy-efficient approach due to its mild operating conditions, shorter extraction times, and reduced need for high temperatures or solvents. However, the exact comparison of energy consumption varies based on several factors, including operating conditions, scale, and efficiency of each extraction method. Future research focusing on maximizing extraction yield, purity, and sustainability while minimizing energy usage and processing costs will be instrumental in advancing green extraction techniques for millet bioactive compounds.

8. Conclusion and future research scope

Millets are a rich source of bioactive compounds, including alkaloids, carotenoids, fatty acids, flavonoids, glycosides, phenolic compounds, phytosterols, saponins, and tannins. Traditional solvent extraction methods often prove costly and energy intensive. Therefore, alternative extraction techniques have been explored to enhance the efficiency of extracting these valuable compounds from millets. Among the alternative green-extraction techniques explored to extract bioactive compounds from millets, ultrasound- and microwave-assisted extraction have shown promise in rapid extraction. Still, there remains a need for more extensive research, particularly in the context of minor millets. One key aspect is the need for further research into optimizing and fine-tuning green extraction methods for millets. This includes exploring the synergistic effects of combining different techniques such as ultrasound, microwave-assisted extraction, and enzyme-assisted extraction to enhance efficiency, yield, and rapid extraction. Investigating the specific parameters that influence extraction outcomes, such as energy efficiency, extraction yield, and compound quality preservation, will be crucial for advancing the field. Additionally, future research should focus on the application of extracted bioactive compounds in developing functional food products and nutraceuticals. Exploring the bioavailability, stability, and potential health benefits of these compounds in millet-based products will be essential for validating the methods for potential application in food and pharmaceutical industries.

Deep eutectic solvents have succeeded in various extraction processes, including those for bioactive compounds from millets.

However, a deeper understanding of their mechanisms and transfer rates is necessary for broader applications. Supercritical fluid extraction holds promise, and assessing the quality, stability, and shelf life of extracted compounds from millets is essential for commercial viability. Combining a single extraction technique with another complementary technique may further enhance the extraction efficiency, necessitating research into optimization and mechanisms. The pulsed electric field is a promising non-thermal tool, but its parameters require further optimization, especially in millets, considering the wide range of bioactive compounds present. Enzyme-assisted extraction offers significant potential, especially when dealing with complex matrices and challenging compounds like millets. Customized protocols, sustainable pre-treatment methods, and exploring new sources can improve effectiveness. Moreover, accelerated solvent extraction has proven effective in extracting bioactive compounds from cereals although there are limited studies. Future research in millet extraction processes should explore synergies between accelerated solvent extraction (ASE) and other methods like ultrasound or microwave assisted extraction (MAE). Investigating these combinations could enhance the efficiency, yield, and speed of bioactive compound extraction. For example, studies could evaluate how ASE combined with ultrasound disrupts cell structures more effectively, leading to increased extraction rates. Similarly, combining ASE with MAE could leverage rapid heating and solvent penetration for improved extraction outcomes. These investigations would contribute valuable insights into optimizing extraction techniques for millets, addressing gaps in current understanding, and paving the way for more efficient and sustainable extraction processes in the food and pharmaceutical industries.

Moreover, there is a growing demand for developing scalable and sustainable extraction processes that can be applied at industrial levels. Scaling up green extraction techniques for millets requires addressing challenges such as process optimization, cost-effectiveness, and compatibility with large-scale production. Further research and optimization are essential to harness their full capabilities. Advanced analytical techniques like mass spectrometry and metabolomics can provide a comprehensive understanding of the extracted compounds. Besides, to scale up the extraction process for industrial applications, further investigation into equipment design, energy efficiency, and process scalability is warranted.

Data availability

Data will be made available on request.

CRediT authorship contribution statement

Nidhi Nayak: Writing – review & editing, Writing – original draft. **Rohan Rajendraji Bhujle:** Writing – review & editing, Writing – original draft. **N.A. Nanje-Gowda:** Writing – review & editing, Writing – original draft, Data curation, Conceptualization. **Snehasis Chakraborty:** Writing – review & editing, Writing – original draft. **Kaliramesh Siliviru:** Writing – review & editing, Writing – original draft, Visualization. **Jeyamkondan Subbiah:** Writing – review & editing, Writing – original draft. **Charles Brennan:** Writing – review & editing, Writing – original draft.

Declaration of competing interest

The authors declare no competing interests associated with the research, ensuring the impartiality and integrity of the presented findings.

References

[1] A. Singh, M. Kumar, M. Shamim, Importance of minor millets (Nutri Cereals) for nutrition purpose in present scenario, Int. J. Chem. Stud. 8 (2020) 3109–3113, https://doi.org/10.22271/chemi.2020.v8.i1au.9226.
[2] F. Silungwe, F. Graef, S. Bellingrath-Kimura, S. Tumbo, F. Kahimba, M. Lana, Analysis of intra and interseasonal rainfall variability and its effects on pearl millet yield in a semiarid agroclimate: significance of scattered fields and tied ridges, Water 11 (2019) 578, https://doi.org/10.3390/w11030578.
[3] M. Thakur, P. Tiwari, Millets: the untapped and underutilized nutritious functional foods, Plant Archives 19 (2019) 875–883.
[4] T. Tamilselvan, S. Sharma, P.E. Thomas, K. Goyal, P. Prabhasankar, Role of hydrocolloids in improving the rheology, quality characteristics, and microstructure of gluten-free proso millet bread, Int. J. Food Sci. Technol. 57 (2022) 7156–7166, https://doi.org/10.1111/ijfs.16058.
[5] S. Dey, A. Saxena, Y. Kumar, T. Maity, A. Tarafdar, Understanding the antinutritional factors and bioactive compounds of kodo millet (paspalum scrobiculatum) and little millet (Panicum sumatrense), J. Food Qual. 2022 (2022) 1–19, https://doi.org/10.1155/2022/1578448.
[6] A.S. Nair, K. Hanumantharaju, A. Lokesh, N.A.N. Gowda, C. Gurikar, Overview on common active compounds present in medicinal plants, Agri 9 (2021) 69–74.
[7] C.K. Sunil, N.A.N. Gowda, N. Nayak, A. Rawson, Unveiling the effect of processing on bioactive compounds in millets: implications for health benefits and risks, Process Biochem. 138 (2024) 79–96, https://doi.org/10.1016/j.procbio.2024.01.010.
[8] T.O. Akanbi, Y. Timilsena, S. Dhital, Bioactives from millet: properties and effects of processing on bioavailability, in: Bioactive Factors and Processing Technology for Cereal Foods, Springer Singapore, Singapore, 2019, pp. 171–183, https://doi.org/10.1007/978-981-13-6167-8_10.
[9] N.A.N. Gowda, K. Siliveru, P.V.V. Prasad, Y. Bhatt, B.P. Netravati, C. Gurikar, Modern processing of Indian millets: a perspective on changes in nutritional properties, Foods 11 (2022) 499, https://doi.org/10.3390/foods11040499.
[10] W. Embashu, K.K.M. Nantanga, Pearl millet grain: a mini-review of the milling, fermentation and brewing of ontaku , a non-alcoholic traditional beverage in Namibia, Trans. Roy. Soc. S. Afr. 74 (2019) 276–282, https://doi.org/10.1080/0035919X.2019.1650310.
[11] B. Zheng, Y. Ding, J.B. Johnson, J. Xiang, Z. Li, Y. Zhang, D. Luo, Enrichment and bioactivities of polyphenols of crude extract by deep eutectic solvent extraction from foxtail millet bran, Int. J. Food Sci. Technol. 57 (2022) 7974–7983, https://doi.org/10.1111/ijfs.16155.
[12] P. Mudgil, M. Alblooshi, B.P. Singh, A.R. Devarajan, S. Maqsood, Pearl millet protein hydrolysates exhibiting effective in-vitro antioxidant, antidiabetic and anti-lipidemic properties as potential functional food ingredient, Int. J. Food Sci. Technol. 58 (2023) 3264–3272, https://doi.org/10.1111/ijfs.16151.
[13] S. Punia Bangar, N. Sharma, A. Singh, Y. Phimolsiripol, C.S. Brennan, Glycaemic response of pseudocereal-based gluten-free food products: a review, Int. J. Food Sci. Technol. 57 (2022) 4936–4944, https://doi.org/10.1111/ijfs.15890.

[14] A.K. Jha, N. Sit, Extraction of bioactive compounds from plant materials using combination of various novel methods: a review, Trends in Food Science {\&} Technology 119 (2022) 579–591, https://doi.org/10.1016/j.tifs.2021.11.019.

[15] N.A.N. Gowda, C. Gurikar, M.B. Anusha, S. Gupta, Ultrasound-assisted and microwave-assisted extraction, GC-MS characterization and antimicrobial potential of freeze-dried L. Camara flower, Journal of Pure and Applied Microbiology 16 (2022) 526–539, https://doi.org/10.22207/JPAM.16.1.50.

[16] K. Kaderides, L. Papaoikonomou, M. Serafim, A.M. Goula, Microwave-assisted extraction of phenolics from pomegranate peels: optimization, kinetics, and comparison with ultrasounds extraction, Chemical Engineering and Processing - Process Intensification 137 (2019) 1–11, https://doi.org/10.1016/j.cep.2019.01.006.

[17] Abert Vian Chemat, Khadhraoui Ravi, Perino Hilali, Tixier, review of alternative solvents for green extraction of food and natural products: panorama, principles, applications and prospects, Molecules 24 (2019) 3007, https://doi.org/10.3390/molecules24163007.

[18] M.B. Soquetta, L. de M. Terra, C.P. Bastos, Green technologies for the extraction of bioactive compounds in fruits and vegetables, CyTA - Journal of Food 16 (2018) 400–412, https://doi.org/10.1080/19476337.2017.1411978.

[19] J. Švarc-Gajić, S. Morais, Recent advances in the development and application of green extraction techniques, Applied Sciences 12 (2022) 10510, https://doi.org/10.3390/app122010510.

[20] B. Zheng, Y. Yuan, J. Xiang, W. Jin, J.B. Johnson, Z. Li, C. Wang, D. Luo, Green extraction of phenolic compounds from foxtail millet bran by ultrasonic-assisted deep eutectic solvent extraction: optimization, comparison and bioactivities, LWT 154 (2022) 112740, https://doi.org/10.1016/j.lwt.2021.112740.

[21] L. Das, M. Li, J. Stevens, W. Li, Y. Pu, A.J. Ragauskas, J. Shi, Characterization and catalytic transfer hydrogenolysis of deep eutectic solvent extracted sorghum lignin to phenolic compounds, ACS Sustainable Chemistry & Engineering 6 (2018) 10408–10420, https://doi.org/10.1021/acssuschemeng.8b01763.

[22] C. Wang, Z. Li, J. Xiang, J. Johnson, B. Zheng, L. Luo, T. Beta, From foxtail millet husk (waste) to bioactive phenolic extracts using deep eutectic solvent extraction and evaluation of antioxidant, acetylcholinesterase, and α-glucosidase inhibitory activities, Foods 12 (2023) 1144, https://doi.org/10.3390/foods12061144.

[23] Y. Shi, Y. Ma, R. Zhang, H. Ma, B. Liu, Preparation and characterization of foxtail millet bran oil using subcritical propane and supercritical carbon dioxide extraction, Journal of Food Science and Technology 52 (2015) 3099–3104, https://doi.org/10.1007/s13197-014-1311-0.

[24] M. Pang, S.J. He, L.L. Cao, S.T. Jiang, Optimization and evaluation of foxtail millet bran oil by supercritical carbon dioxide extraction, Grasas y Aceites 66 (2015) e107, https://doi.org/10.3989/gya.0239151.

[25] A. Tuhanioglu, A. Ubeyitogullari, Extraction of high-value lipids and phenolic compounds from sorghum bran via a sequential supercritical carbon dioxide approach, ACS Food Science & Technology 2 (2022) 1879–1887, https://doi.org/10.1021/acsfoodscitech.2c00266.

[26] F. Sperotto, J. Yang, L. Isom, C. Weller, O.N. Ciftci, Supercritical carbon dioxide extraction, purification, and characterization of wax from sorghum and sorghum by-products as an alternative natural wax, Journal of the American Oil Chemists' Society 99 (2022) 433–441, https://doi.org/10.1002/aocs.12575.

[27] L. Wang, C.L. Weller, V.L. Schlegel, T.P. Carr, S.L. Cuppett, Comparison of supercritical CO2 and hexane extraction of lipids from sorghum distillers grains, European Journal of Lipid Science and Technology 109 (2007) 567–574, https://doi.org/10.1002/ejlt.200700018.

[28] L. Wang, C.L. Weller, V.L. Schlegel, T.P. Carr, S.L. Cuppett, Comparison of supercritical CO_2 and hexane extraction of lipids from sorghum distillers grains, European Journal of Lipid Science and Technology 109 (2007) 567–574, https://doi.org/10.1002/ejlt.200700018.

[29] U.C. Lohani, K. Muthukumarappan, Study of continuous flow ultrasonication to improve total phenolic content and antioxidant activity in sorghum flour and its comparison with batch ultrasonication, Ultrasonics Sonochemistry 71 (2021) 105402, https://doi.org/10.1016/j.ultsonch.2020.105402.

[30] F. Hou, D. Su, J. Xu, Y. Gong, R. Zhang, Z. Wei, J. Chi, M. Zhang, Enhanced extraction of phenolics and antioxidant capacity from sorghum (iSorghum bicolor L/i . Moench) shell using ultrasonic-assisted ethanol-water binary solvent, Journal of Food Processing and Preservation 40 (2016) 1171–1179, https://doi.org/10.1111/jfpp.12699.

[31] X. Luo, J. Cui, H. Zhang, Y. Duan, D. Zhang, M. Cai, G. Chen, Ultrasound assisted extraction of polyphenolic compounds from red sorghum (Sorghum bicolor L.) bran and their biological activities and polyphenolic compositions, Industrial Crops and Products 112 (2018) 296–304, https://doi.org/10.1016/j.indcrop.2017.12.019.

[32] S. Hassan, M. Imran, M.H. Ahmad, M.I. Khan, C. Xu, M.K. Khan, N. Muhammad, Phytochemical characterization of ultrasound-processed sorghum sprouts for the use in functional foods, International Journal of Food Properties 23 (2020) 853–863, https://doi.org/10.1080/10942912.2020.1762644.

[33] N. Čukelj Mustač, D. Novotni, M. Habuš, S. Drakula, L. Nanjara, B. Voučko, M. Benković, D. Ćurić, Storage stability, micronisation, and application of nutrient-dense fraction of proso millet bran in gluten-free bread, Journal of Cereal Science 91 (2020) 102864, https://doi.org/10.1016/j.jcs.2019.102864.

[34] S. Shukla, U.C. Lohani, N.C. Shahi, A. Dubey, Extraction of natural pigments from red sorghum (Sorghum bicolor) husk by ultrasound and microwave assisted extraction: a comparative study through response surface analysis, Journal of Food Process Engineering 45 (2022) e14130, https://doi.org/10.1111/jfpe.14130.

[35] D.A. Herrman, J.F. Brantsen, S. Ravisankar, K.-M. Lee, J.M. Awika, Stability of 3-deoxyanthocyanin pigment structure relative to anthocyanins from grains under microwave assisted extraction, Food Chemistry 333 (2020) 127494, https://doi.org/10.1016/j.foodchem.2020.127494.

[36] C. Chiremba, L.W. Rooney, T. Beta, Microwave-assisted extraction of bound phenolic acids in bran and flour fractions from sorghum and maize cultivars varying in hardness, Journal of Agricultural and Food Chemistry 60 (2012) 4735–4742, https://doi.org/10.1021/jf300279t.

[37] K. Duke, C. Syeunda, J.F. Brantsen, S. Nindawat, J. Awika, Polyphenol recovery from sorghum bran waste by microwave assisted extraction: structural transformations as affected by grain phenolic profile, SSRN (2022) 1–30. https://ssrn.com/abstract=4266969.

[38] S. Palaniswamy, Development of a Millet Dehuller (Hand-operated) to Reduce Drudgery in Processing and Utilization of Milet Waste (Hulls) in Antioxidant Extraction, McGill University (Canada), 2018. https://www.proquest.com/openview/821636654ae8df212646185f9bd84cce/1?pq-origsite=gscholar&cbl=18750&diss=y.

[39] A. Siddiq A, J. Prakash, Antioxidant properties of digestive enzyme-treated fibre-rich fractions from wheat, finger millet, pearl millet and sorghum: a comparative evaluation, Cogent Food & Agriculture 1 (2015) 1073875, https://doi.org/10.1080/23311932.2015.1073875.

[40] H. Agrawal, R. Joshi, M. Gupta, Isolation, purification and characterization of antioxidative peptide of pearl millet (Pennisetum glaucum) protein hydrolysate, Food Chemistry 204 (2016) 365–372, https://doi.org/10.1016/j.foodchem.2016.02.127.

[41] V.G. Balasubramaniam, P. Ayyappan, S. Sathvika, U. Antony, Effect of enzyme pretreatment in the ultrasound assisted extraction of finger millet polyphenols, Journal of Food Science and Technology 56 (2019) 1583–1594, https://doi.org/10.1007/s13197-019-03672-2.

[42] G. Yadav, A. Singh, P. Bhattacharya, J. Yuvraj, R. Banerjee, Comparative analysis of solid-state bioprocessing and enzymatic treatment of finger millet for mobilization of bound phenolics, Bioprocess and Biosystems Engineering 36 (2013) 1563–1569, https://doi.org/10.1007/s00449-013-0924-4.

[43] H. Agrawal, R. Joshi, M. Gupta, Isolation and characterisation of enzymatic hydrolysed peptides with antioxidant activities from green tender sorghum, LWT 84 (2017) 608–616, https://doi.org/10.1016/j.lwt.2017.06.036.

[44] E. Zong, D. Guo, S. Fan, B. Bai, J. Zhang, Optimization of the Extraction Process of Millet Bran Polysaccharides and Antioxidant Activities Analysis, Researchsquare, 2022, pp. 1–25, https://doi.org/10.21203/rs.3.rs-2138967/v1.

[45] M.A. Kurek, S. Karp, J. Wyrwisz, Y. Niu, Physicochemical properties of dietary fibers extracted from gluten-free sources: quinoa (Chenopodium quinoa), amaranth (Amaranthus caudatus) and millet (Panicum miliaceum), Food Hydrocolloids 85 (2018) 321–330, https://doi.org/10.1016/j.foodhyd.2018.07.021.

[46] F. Barros, L. Dykes, J.M. Awika, L.W. Rooney, Accelerated solvent extraction of phenolic compounds from sorghum brans, Journal of Cereal Science 58 (2013) 305–312, https://doi.org/10.1016/j.jcs.2013.05.011.

[47] M. Samtiya, R.E. Aluko, N. Dhaka, T. Dhewa, A.K. Puniya, Nutritional and health-promoting attributes of millet: current and future perspectives, Nutrition Reviews 81 (2023) 684–704, https://doi.org/10.1093/nutrit/nuac081.

[48] Z. Li, X. Zhao, X. Zhang, H. Liu, The effects of processing on bioactive compounds and biological activities of sorghum grains, Molecules 27 (2022), https://doi.org/10.3390/molecules27103246.

[49] H. Davis, X. Su, Y. Shen, J. Xu, D. Wang, J. Scott Smith, F. Aramouni, W. Wang, Phenotypic diversity of colored phytochemicals in sorghum accessions with various pericarp pigments, in: Polyphenols in Plants, Elsevier, 2019, pp. 123–131, https://doi.org/10.1016/B978-0-12-813768-0.00008-6.

[50] D. Mounika, U. Sangeetha, G. Sireesha, Estimation of phytochemicals in Millets and selected Millet products, Indian J. Appl. Pure Biology. 37 (2022) 810–820. https://www.researchgate.net/profile/Sireesha-Guttapalam/publication/363609161_Estimation_of_phytochemicals_in_Millets_and_selected_Millet_products/links/6324a7740a70852150fb55e0/Estimation-of-phytochemicals-in-Millets-and-selected-Millet-products.pdf.

[51] S. Liang, K. Liang, Millet grain as a candidate antioxidant food resource: a review, International Journal of Food Properties 22 (2019), https://doi.org/10.1080/10942912.2019.1668406.

[52] A. Slama, A. Cherif, F. Sakouhi, S. Boukhchina, L. Radhouane, Fatty acids, phytochemical composition and antioxidant potential of pearl millet oil, Journal of Consumer Protection and Food Safety 15 (2020) 145–151, https://doi.org/10.1007/s00003-019-01250-4.

[53] F. Shahidi, R. Danielski, C. Ikeda, Phenolic compounds in cereal grains and effects of processing on their composition and bioactivities: a review, Journal of Food Bioactives 15 (2021), https://doi.org/10.31665/JFB.2021.15281.

[54] J. Xiang, F.B. Apea-Bah, V.U. Ndolo, M.C. Katundu, T. Beta, Profile of phenolic compounds and antioxidant activity of finger millet varieties, Food Chemistry 275 (2019) 361–368, https://doi.org/10.1016/j.foodchem.2018.09.120.

[55] S. Mitharwal, S. Kumar, K. Chauhan, Nutritional, polyphenolic composition and in vitro digestibility of finger millet (Eleusine coracana L.) with its potential food applications: a review, Food Bioscience 44 (2021) 101382, https://doi.org/10.1016/j.fbio.2021.101382.

[56] P. Shah, A. Kumar, V. Kumar, M.K. Tripathi, Millets, phytochemicals, and their health attributes, in: Millets and Millet Technology, Springer Singapore, Singapore, 2021, pp. 191–218, https://doi.org/10.1007/978-981-16-0676-2_9.

[57] T.H.D. Nguyen, L.L.P. Nguyen, L. Baranyai, Influence of sprouting on phenolic acids, carotenoids, and antioxidant activity of millet varieties, Journal of Agriculture and Food Research 14 (2023) 100810, https://doi.org/10.1016/j.jafr.2023.100810.

[58] S. Yadav, Efficacy of kodo millets paspalum scrobiculatum: a systematic review, Bioscience Biotechnology Research Communications 15 (2022) 379–385, https://doi.org/10.21786/bbrc/15.3.2.

[59] A. Singh, M. Bharath, A. Kotiyal, L. Rana, D. Rajpal, Barnyard millet: the underutilized nutraceutical minor millet crop, J Pharm Innov 11 (2022) 115–128.

[60] S. Kumari, S. Bhinder, B. Singh, A. Kaur, Physicochemical properties, non-nutrients and phenolic composition of germinated freeze-dried flours of foxtail millet, proso millet and common buckwheat, Journal of Food Composition and Analysis 115 (2023) 105043, https://doi.org/10.1016/j.jfca.2022.105043.

[61] R. Sharma, S. Sharma, B. Singh, Modulation in the bio-functional & technological characteristics, in vitro digestibility, structural and molecular interactions during bioprocessing of proso millet (Panicum miliaceum L.), Journal of Food Composition and Analysis 107 (2022) 104372, https://doi.org/10.1016/j.jfca.2021.104372.

[62] U.B. Akuru, B.A. Amadi, Phytochemicals and antioxidant properties of some selected medicinal plants, Journal of Pharmacognosy and Phytochemistry 7 (2018) 283–285.

[63] P. Panwar, A. Dubey, A.K. Verma, Evaluation of nutraceutical and antinutritional properties in barnyard and finger millet varieties grown in Himalayan region, Journal of Food Science and Technology 53 (2016) 2779–2787, https://doi.org/10.1007/s13197-016-2250-8.

[64] R. Kharsan, D.K.M. Panjwani, Phytochemical evaluation of Kodo (Paspalum scrobiculatum L.) bran, Journal of Research in Chemistry. 4 (2023) 26–30.

[65] L. Vandana, In-vitro study for evaluation of proximate composition, phytochemical & neutraceutical properties of different millet samples, International Journal of Science and Research (IJSR) 7 (2018) 1889–1892. https://www.ijsr.net/archive/v7i11/ART20192921.pdf.

[66] K.S. Woo, J.S. Lee, J.Y. Ko, S.B. Song, H.I. Seo, M.C. Seo, B.G. Oh, D.Y. Kwak, M.H. Nam, I.S. Oh, H.S. Jeong, Antioxidant compounds and antioxidant activities of different varieties of foxtail millet and proso millet according to cultivation time, Journal of the Korean Society of Food Science and Nutrition 41 (2012) 302–309, https://doi.org/10.3746/jkfn.2012.41.3.302.

[67] F.S. Pushparaj, A. Urooj, Influence of processing on dietary fiber, tannin and in vitro protein digestibility of pearl millet, Food and Nutrition Sciences 2 (2011) 895–900.

[68] M. Sorour, A. Mehanni, E. Taha, A. Rashwan, Changes of total phenolics, tannins, phytate and antioxidant activity of two sorghum cultivars as affected by processing, Journal of Food and Dairy Sciences 8 (2017) 267–274, https://doi.org/10.21608/jfds.2017.38699.

[69] Y. Yuan, J. Xiang, B. Zheng, J. Sun, D. Luo, P. Li, J. Fan, Diversity of phenolics including hydroxycinnamic acid amide derivatives, phenolic acids contribute to antioxidant properties of proso millet, Lwt 154 (2022) 112611.

[70] S. Sharma, D.C. Saxena, C.S. Riar, Antioxidant activity, total phenolics, flavonoids and antinutritional characteristics of germinated foxtail millet (Setaria italica), Cogent Food and Agriculture (2015), https://doi.org/10.1080/23311932.2015.1081728.

[71] S. Jevcsák, E. Murányi, L. Stündl, J. Jóvér, P. Sipos, Effect of different levels of nitrogen on the total polyphenol and total flavonoid content of sorghum and millet flours, Acta Universitatis Sapientiae, Alimentaria 10 (2017) 107–115, https://doi.org/10.1515/ausal-2017-0007.

[72] G. Goudar, M.C. Khetagoudar, S.R. Patil, Evaluation of hydroxycinnamic acids in finger millet (Eleusine coracana) grain cultivars by HPTLC, $~$ 328, International Journal of Chemical Studies. 6 (2018) 328–332.

[73] P.M. Pradeep, Y.N. Sreerama, Phenolic antioxidants of foxtail and little millet cultivars and their inhibitory effects on $α$-amylase and $α$-glucosidase activities, Food Chemistry 247 (2018) 46–55, https://doi.org/10.1016/j.foodchem.2017.11.103.

[74] R. Devisetti, S.N. Yadahally, S. Bhattacharya, Nutrients and antinutrients in foxtail and proso millet milled fractions: evaluation of their flour functionality, LWT - Food Science and Technology 59 (2014) 889–895, https://doi.org/10.1016/j.lwt.2014.07.003.

[75] A.K. Siroha, K.S. Sandhu, M. Kaur, Physicochemical, functional and antioxidant properties of flour from pearl millet varieties grown in India, Journal of Food Measurement and Characterization 10 (2016) 311–318, https://doi.org/10.1007/s11694-016-9308-1.

[76] I. Pasha, A. Riaz, M. Saeed, M.A. Randhawa, Exploring the antioxidant perspective of sorghum and millet, Journal of Food Processing and Preservation 39 (2015) 1089–1097, https://doi.org/10.1111/jfpp.12323.

[77] U. Srivastava, P. Saini, A. Singh, Z. Singh, M. Ahmed, U. Iqbal, Enhancement in iron and folate by optimizing fermentation of barnyard millet by lactobacillus plantarum using response surface methodology (rsm), Plant Archives 21 (2021) 993–1005.

[78] R. Upadhyay, A. Jha, S.P. Singh, A. Kumar, M. Singh, Appropriate solvents for extracting total phenolics, flavonoids and ascorbic acid from different kinds of millets, Journal of Food Science and Technology 52 (2015) 472–478, https://doi.org/10.1007/s13197-013-0976-0.

[79] S.C. Choi, J.M. Kim, Y.G. Lee, C. Kim, Antioxidant activity and contents of total phenolic compounds and anthocyanins according to grain colour in several varieties of sorghum bicolor (L.) moench, Cereal Research Communications 47 (2019) 228–238, https://doi.org/10.1556/0806.47.2019.14.

[80] M. Mutshinyani, M.E. Mashau, A.I.O. Jideani, Bioactive compounds, antioxidant activity and consumer acceptability of porridges of finger millet (Eleusine coracana) flours: effects of spontaneous fermentation, International Journal of Food Properties 23 (2020) 1692–1710, https://doi.org/10.1080/10942912.2020.1825485.

[81] V.T. Asharani, A. Jayadeep, N.G. Malleshi, Natural antioxidants in edible flours of selected small millets, International Journal of Food Properties 13 (2010) 41–50, https://doi.org/10.1080/10942910802163105.

[82] B.S. Khangura, K.S. Gill, P.S. Phul, Combining ability analysis of beta-Carotene, total carotenoids and other grain characteristics in pearl millet, Theoretical and Applied Genetics 56 (1980) 91–96, https://doi.org/10.1007/BF00264433.

[83] M.G.S. Fernandez, M.T. Hamblin, L. Li, W.L. Rooney, M.R. Tuinstra, S. Kresovich, Quantitative trait loci analysis of endosperm color and carotenoid content in sorghum grain, Crop Science 48 (2008) 1732–1743, https://doi.org/10.2135/cropsci2007.12.0684.

[84] R. Salvador-Reyes, M.T.P.S. Clerici, Peruvian Andean maize: general characteristics, nutritional properties, bioactive compounds, and culinary uses, Food Research International 130 (2020) 108934, https://doi.org/10.1016/j.foodres.2019.108934.

[85] C. Gunathunga, S. Senanayake, M.A. Jayasinghe, C.S. Brennan, T. Truong, U. Marapana, J. Chandrapala, Germination effects on nutritional quality: a comprehensive review of selected cereals and pulses changes, Journal of Food Composition and Analysis 128 (2024) 106024, https://doi.org/10.1016/j.jfca.2024.106024.

[86] I.D. Boateng, R. Kumar, C.R. Daubert, S. Flint-Garcia, A. Mustapha, L. Kuehnel, J. Agliata, Q. Li, C. Wan, P. Somavat, Sonoprocessing improves phenolics profile, antioxidant capacity, structure, and product qualities of purple corn pericarp extract, Ultrasonics Sonochemistry 95 (2023) 106418, https://doi.org/10.1016/j.ultsonch.2023.106418.

[87] R. Gupta, M. Meghwal, P.K. Prabhakar, Bioactive compounds of pigmented wheat (Triticum aestivum): potential benefits in human health, Trends in Food Science {\&} Technology 110 (2021) 240–252, https://doi.org/10.1016/j.tifs.2021.02.003.

[88] J. Liu, L.L. Yu, Y. Wu, Bioactive components and health beneficial properties of whole wheat foods, Journal of Agricultural and Food Chemistry 68 (2020) 12904–12915, https://doi.org/10.1021/acs.jafc.0c00705.

[89] D.K. Verma, P.P. Srivastav, Bioactive compounds of rice (Oryza sativa L.): review on paradigm and its potential benefit in human health, Trends in Food Science & Technology 97 (2020) 355–365, https://doi.org/10.1016/j.tifs.2020.01.007.

[90] T. Xiu, P. Liu, S. Zhang, D. Du, C. Xue, Y. Hu, S. Yang, Z. Dongye, M. Kang, Z. Li, Polyphenol nanoparticles of millet, rice and wheat: extraction, identification, functional and morphological characteristics, International Journal of Food Science & Technology 57 (2022) 4973–4984.

[91] S.A. Sofi, Cereal bioactive compounds: a review, International Journal of Agriculture Environment and Biotechnology 12 (2019) 107–113, https://doi.org/10.30954/0974-1712.06.2019.5.

[92] R.S. Arsha, P. Rasane, J. Singh, Rice: bioactive compounds and their health benefits, The Pharma Innovation Journal 10 (2021) 845–853.

[93] M. Obadi, J. Sun, B. Xu, Highland barley: chemical composition, bioactive compounds, health effects, and applications, Food Research International 140 (2021) 110065, https://doi.org/10.1016/j.foodres.2020.110065.

[94] Y. Zeng, X. Pu, J. Du, X. Yang, X. Li, M.S.N. Mandal, T. Yang, J. Yang, Molecular mechanism of functional ingredients in barley to combat human chronic diseases, Oxidative Medicine and Cellular Longevity 2020 (2020) 1–26, https://doi.org/10.1155/2020/3836172.

[95] Y. Zhang, Y. Lin, L. Huang, M. Tekliye, H.A. Rasheed, M. Dong, Composition, antioxidant, and anti-biofilm activity of anthocyanin-rich aqueous extract from purple highland barley bran, LWT 125 (2020) 109181, https://doi.org/10.1016/j.lwt.2020.109181.

[96] N. Mäkelä, N. Rosa-Sibakov, Y.-J. Wang, O. Mattila, E. Nordlund, T. Sontag-Strohm, Role of β-glucan content, molecular weight and phytate in the bile acid binding of oat β-glucan, Food Chemistry 358 (2021) 129917, https://doi.org/10.1016/j.foodchem.2021.129917.

[97] P. Vadim, L. Igor, S. Alena, Biological role and health benefits of antioxidant compounds in cereals, Biological Communications 65 (2020) 53–67.

[98] D. Paudel, B. Dhungana, M. Caffe, P. Krishnan, A review of health-beneficial properties of oats, Foods 10 (2021) 2591, https://doi.org/10.3390/foods10112591.

[99] M.A. Islam, B.-G. Jeong, J. Jung, E.-C. Shin, S.-G. Choi, J. Chun, Phytosterol determination and method validation for selected nuts and seeds, Food Analytical Methods 10 (2017) 3225–3234, https://doi.org/10.1007/s12161-017-0877-3.

[100] B. Hernández-Ledesma, Quinoa (Chenopodium Quinoa Willd.) as Source of Bioactive Compounds: a Review, Bioactive Compounds in Health and Disease, vol. 2, 2019, p. 27, https://doi.org/10.31989/bchd.v2i3.556.

[101] H. Li, Buckwheat, in: Bioactive Factors and Processing Technology for Cereal Foods, Springer Singapore, Singapore, 2019, pp. 137–149, https://doi.org/10.1007/978-981-13-6167-8_8.

[102] M.N. Huda, S. Lu, T. Jahan, M. Ding, R. Jha, K. Zhang, W. Zhang, M.I. Georgiev, S.U. Park, M. Zhou, Treasure from garden: bioactive compounds of buckwheat, Food Chemistry 335 (2021) 127653, https://doi.org/10.1016/j.foodchem.2020.127653.

[103] S.A. Borovaya, A.G. Klykov, Some aspects of flavonoid biosynthesis and accumulation in buckwheat plants, Plant Biotechnology Reports 14 (2020) 213–225, https://doi.org/10.1007/s11816-020-00614-9.

[104] R.E. Jocelyne, K. Béhiblo, A.K. Ernest, Comparative study of nutritional value of wheat, maize, sorghum, millet, and fonio: some cereals commonly consumed in côte d'Ivoire, European Scientific Journal ESJ 16 (2020) 118–131, https://doi.org/10.19044/esj.2020.v16n21p118.

[105] U. Sarker, S. Oba, S. Ercisli, A. Assouguem, A. Alotaibi, R. Ullah, Bioactive phytochemicals and quenching activity of radicals in selected drought-resistant Amaranthus tricolor vegetable amaranth, Antioxidants 11 (2022) 578, https://doi.org/10.3390/antiox11030578.

[106] M. Tomar, R. Bhardwaj, R. Verma, S.P. Singh, A. Dahuja, V. Krishnan, R. Kansal, V.K. Yadav, S. Praveen, A. Sachdev, Interactome of millet-based food matrices: a review, Food Chemistry 385 (2022) 132636, https://doi.org/10.1016/j.foodchem.2022.132636.

[107] M. Garcia-Vaquero, G. Rajauria, B. Tiwari, Conventional extraction techniques: solvent extraction, in: Sustainable Seaweed Technologies, Elsevier, 2020, pp. 171–189, https://doi.org/10.1016/B978-0-12-817943-7.00006-8.

[108] G. Li, L. Ma, Z. Yan, Q. Zhu, J. Cai, S. Wang, Y. Yuan, Y. Chen, S. Deng, Extraction of oils and phytochemicals from camellia oleifera seeds: trends, challenges, and innovations, Processes 10 (2022) 1489, https://doi.org/10.3390/pr10081489.

[109] S.A. Moreira, E.M.C. Alexandre, M. Pintado, J.A. Saraiva, Effect of emergent non-thermal extraction technologies on bioactive individual compounds profile from different plant materials, Food Research International 115 (2019) 177–190, https://doi.org/10.1016/j.foodres.2018.08.046.

[110] S. Satriana, M.D. Supardan, N. Arpi, W.A. Wan Mustapha, Development of methods used in the extraction of avocado oil, European Journal of Lipid Science and Technology 121 (2019) 1800210, https://doi.org/10.1002/ejlt.201800210.

[111] M.A. Aitkazina, N. Ermek, S. Syrlybekkyzy, S. Koibakova, A.E. Zhidebayeva, M.Z. Aubakiro, Threats to sustainable development due to increase of greenhouse gas emissions in a key sector, Journal of Security and Sustainability Issues. 9 (17) (2019) 227–240, https://doi.org/10.9770/jssi.2019.9.1.

[112] J. Chen, Y. Li, X. Wang, W. Liu, Application of deep eutectic solvents in food analysis: a review, Molecules 24 (2019) 4594, https://doi.org/10.3390/molecules24244594.

[113] N. Bhargava, R.S. Mor, K. Kumar, V.S. Sharanagat, Advances in application of ultrasound in food processing: a review, Ultrasonics Sonochemistry 70 (2021), https://doi.org/10.1016/j.ultsonch.2020.105293.

[114] T. Sireesha, N.A.N. Gowda, V. Kambhampati, Ultrasonication in seafood processing and preservation: a comprehensive review, Applied Food Research 2 (2022) 100208, https://doi.org/10.1016/j.afres.2022.100208.

[115] N.Q.I.M. Noor, R.S. Razali, N.K. Ismail, R.A. Ramli, U.H.M. Razali, A.R. Bahauddin, N. Zaharudin, A. Rozzamri, J. Bakar, S.M. Shaarani, Application of green technology in gelatin extraction: a review, Processes 9 (2021) 2227, https://doi.org/10.3390/pr9122227.

[116] M.V. Rao, A.S. Sengar, S. C K, A. Rawson, Ultrasonication - a green technology extraction technique for spices: a review, Trends in Food Science {\&} Technology 116 (2021) 975–991, https://doi.org/10.1016/j.tifs.2021.09.006.

[117] P. Wu, X. Wang, W. Lin, L. Bai, Acoustic characterization of cavitation intensity: a review, Ultrasonics Sonochemistry 82 (2022) 105878, https://doi.org/10.1016/j.ultsonch.2021.105878.

[118] D. Schieppati, M. Mohan, B. Blais, K. Fattahi, G.S. Patience, B.A. Simmons, S. Singh, D.C. Boffito, Characterization of the acoustic cavitation in ionic liquids in a horn-type ultrasound reactor, Ultrasonics Sonochemistry 102 (2024) 106721, https://doi.org/10.1016/j.ultsonch.2023.106721.

[119] L. Meena, N.N. Gowda, C.K. Sunil, A. Rawson, S. Janghu, Effect of ultrasonication on food bioactive compounds and their bio-accessibility: a review, Journal of Food Composition and Analysis 126 (2024) 105899, https://doi.org/10.1016/j.jfca.2023.105899.

[120] T. Wu, J. Yan, R. Liu, M.F. Marcone, H.A. Aisa, R. Tsao, Optimization of microwave-assisted extraction of phenolics from potato and its downstream waste using orthogonal array design, Food Chemistry 133 (2012) 1292–1298, https://doi.org/10.1016/j.foodchem.2011.08.002.

[121] C. Carrera, A. Ruiz-Rodríguez, M. Palma, C.G. Barroso, Ultrasound assisted extraction of phenolic compounds from grapes, Analytica Chimica Acta 732 (2012) 100–104, https://doi.org/10.1016/j.aca.2011.11.032.

[122] N.Č. Mustač, B. Voučko, D. Novotni, S. Drakula, A. Gudelj, F. Dujmić, D. Ćurić, F. Dujmić, A. Gudelj, S. Drakula, B. Voučko, N. Čukelj Mustač, Optimization of high intensity ultrasound treatment of proso millet bran to improve physical and nutritional quality, Food Technology and Biotechnology 57 (2019) 183–190, https://doi.org/10.17113/ftb.57.02.19.6100.

[123] Q. Wang, G. Chen, H. Yersaiyiti, Y. Liu, J. Cui, C. Wu, Y. Zhang, X. He, Modeling analysis on germination and seedling growth using ultrasound seed pretreatment in switchgrass, PLoS ONE 7 (2012) e47204, https://doi.org/10.1371/journal.pone.0047204.

[124] J. Liu, Q. Wang, Đ. Karagić, X. Liu, J. Cui, J. Gui, M. Gu, W. Gao, Effects of ultrasonication on increased germination and improved seedling growth of aged grass seeds of tall fescue and Russian wildrye, Scientific Reports 6 (2016) 22403, https://doi.org/10.1038/srep22403.

[125] S.N. Raj, B.R. Sarosh, H.S. Shetty, Induction and accumulation of polyphenol oxidase activities as implicated in development of resistance against pearl millet downy mildew disease, Functional Plant Biology 33 (2006) 563, https://doi.org/10.1071/FP06003.

[126] Y. Wang, Y. Hu, H. Wang, M. Tong, Y. Gong, Green and enhanced extraction of coumarins from iCortex Fraxini/i by ultrasound-assisted deep eutectic solvent extraction, Journal of Separation Science 43 (2020) 3441–3448, https://doi.org/10.1002/jssc.202000334.

[127] K. Bhatt, D. Vaidya, M. Kaushal, A. Gupta, P. Soni, P. Arya, A. Gautam, C. Sharma, Microwaves and radiowaves: in food processing and preservation, International Journal of Current Microbiology and Applied Sciences 9 (2020) 118–131.
[128] O. Altin, D. Skipnes, T. Skåra, F. Erdogdu, A computational study for the effects of sample movement and cavity geometry in industrial scale continuous microwave systems during heating and thawing processes, Innovative Food Science & Emerging Technologies 77 (2022) 102953, https://doi.org/10.1016/j.ifset.2022.102953.
[129] M. Peng, W. Li, H. Tan, L. Lin, Simulation study on microwave heating mechanism of shale oil in situ, in: Proceedings of the International Petroleum and Petrochemical Technology Conference 2020, Springer Singapore, Singapore, 2021, pp. 439–455, https://doi.org/10.1007/978-981-16-1123-0_42.
[130] R. Zou, X. Zhou, M. Qian, C. Wang, D. Boldor, H. Lei, X. Zhang, Advancements and applications of microwave-assisted deep eutectic solvent (MW-DES) lignin extraction: a comprehensive review, Green Chemistry 26 (2024) 1153–1169, https://doi.org/10.1039/D3GC04501C.
[131] A. Plaza, L. Rodríguez, A.A. Concha-Meyer, R. Cabezas, E. Zurob, G. Merlet, I. Palomo, E. Fuentes, Effects of extraction methods on phenolic content, antioxidant and antiplatelet activities of tomato pomace extracts, Plants 12 (2023) 1188, https://doi.org/10.3390/plants12051188.
[132] Y. Umama, M.K. Gayoor, V. G, R. Shourabh, K. Roshan, A. Verma, A. Kumar, The scenario of pharmaceuticals and development of microwave assisted extraction techniques, SSRN Electronic Journal 8 (2019) 1260–1271, https://doi.org/10.2139/ssrn.3557317.
[133] J.O. Chaves, M.C. de Souza, L.C. da Silva, D. Lachos-Perez, P.C. Torres-Mayanga, A.P. da F. Machado, T. Forster-Carneiro, M. Vázquez-Espinosa, A.V. González-de-Peredo, G.F. Barbero, M.A. Rostagno, Extraction of flavonoids from natural sources using modern techniques, Frontiers in Chemistry 8 (2020) 507887, https://doi.org/10.3389/fchem.2020.507887.
[134] P. Skenderidis, S. Leontopoulos, K. Petrotos, I. Giavasis, Optimization of vacuum microwave-assisted extraction of pomegranate fruits peels by the evaluation of extracts' phenolic content and antioxidant activity, Foods 9 (2020) 1655, https://doi.org/10.3390/foods9111655.
[135] R. Li, Q. Wang, H. Peng, G. Zhao, D. Zhang, Z. Li, Exploring the effect of microwave treatment on phenolic flavonoids, antioxidant capacity, and phenolic in vitro bioaccessibility of sorghum, International Journal of Food Science & Technology 57 (2022) 2510–2522, https://doi.org/10.1111/ijfs.15624.
[136] E.M. Abdel-Aal, H. Akhtar, I. Rabalski, M. Bryan, Accelerated, microwave-assisted, and conventional solvent extraction methods affect anthocyanin composition from colored grains, Journal of Food Science 79 (2014) C138–C146, https://doi.org/10.1111/1750-3841.12346.
[137] S.R. Shirsath, S.H. Sonawane, P.R. Gogate, Intensification of extraction of natural products using ultrasonic irradiations- A review of current status, Chemical Engineering and Processing: Process Intensification 53 (2012) 10–23, https://doi.org/10.1016/j.cep.2012.01.003.
[138] V. Gude, P. Patil, E. Martinez-Guerra, S. Deng, N. Nirmalakhandan, Microwave energy potential for biodiesel production, Sustainable Chemical Processes 1 (2013) 5, https://doi.org/10.1186/2043-7129-1-5.
[139] A.B. Rabah, Utilization of millet and Guinea corn husks for bioethanol production, African Journal of Microbiology Research 5 (2011) 5721–5724, https://doi.org/10.5897/AJMR11.1127.
[140] M. Zhang, Y. Xu, J. Xiang, B. Zheng, Y. Yuan, D. Luo, J. Fan, Comparative evaluation on phenolic profiles, antioxidant properties and alpha-glucosidase inhibitory effects of different milling fractions of foxtail millet, Journal of Cereal Science 99 (2021) 103217, https://doi.org/10.1016/j.jcs.2021.103217.
[141] Z. Yang, W. Zhai, Identification and antioxidant activity of anthocyanins extracted from the seed and cob of purple corn (Zea mays L.), Innovative Food Science & Emerging Technologies 11 (2010) 169–176, https://doi.org/10.1016/j.ifset.2009.08.012.
[142] J. Wizi, L. Wang, X. Hou, Y. Tao, B. Ma, Y. Yang, Ultrasound-microwave assisted extraction of natural colorants from sorghum husk with different solvents, Industrial Crops and Products 120 (2018) 203–213, https://doi.org/10.1016/j.indcrop.2018.04.068.
[143] M. Vázquez-Espinosa, A.V.G. de Peredo, M. Ferreiro-González, C. Carrera, M. Palma, G.F. Barbero, E. Espada-Bellido, Assessment of ultrasound assisted extraction as an alternative method for the extraction of anthocyanins and total phenolic compounds from maqui berries (aristotelia chilensis (mol.) stuntz), Agronomy 9 (2019) 148, https://doi.org/10.3390/agronomy9030148.
[144] E. Roselló-Soto, C.M. Galanakis, M. Brnčić, V. Orlien, F.J. Trujillo, R. Mawson, K. Knoerzer, B.K. Tiwari, F.J. Barba, Clean recovery of antioxidant compounds from plant foods, by-products and algae assisted by ultrasounds processing. Modeling approaches to optimize processing conditions, Trends in Food Science & Technology 42 (2015) 134–149, https://doi.org/10.1016/j.tifs.2015.01.002.
[145] F.M. Perna, P. Vitale, V. Capriati, Deep eutectic solvents and their applications as green solvents, Current Opinion in Green and Sustainable Chemistry 21 (2020) 27–33, https://doi.org/10.1016/j.cogsc.2019.09.004.
[146] K. Zhang, C. Liu, S. Li, J. Fan, A hydrophobic deep eutectic solvent based vortex-assisted liquid-liquid microextraction for the determination of formaldehyde from biological and indoor air samples by high performance liquid chromatography, Journal of Chromatography A 1589 (2019) 39–46, https://doi.org/10.1016/j.chroma.2018.12.063.
[147] D. Chandran, M. Khalid, R. Walvekar, N.M. Mubarak, S. Dharaskar, W.Y. Wong, T.C.S.M. Gupta, Deep eutectic solvents for extraction-desulphurization: a review, Journal of Molecular Liquids 275 (2019) 312–322, https://doi.org/10.1016/j.molliq.2018.11.051.
[148] Z. Usmani, M. Sharma, M. Tripathi, T. Lukk, Y. Karpichev, N. Gathergood, B.N. Singh, V.K. Thakur, M. Tabatabaei, V.K. Gupta, Biobased natural deep eutectic system as versatile solvents: structure, interaction and advanced applications, Science of The Total Environment 881 (2023) 163002, https://doi.org/10.1016/j.scitotenv.2023.163002.
[149] N. Mustafa, V. Spelbos, G.-J. Witkamp, R. Verpoorte, Y. Choi, Solubility and stability of some pharmaceuticals in natural deep eutectic solvents-based formulations, Molecules 26 (2021) 2645, https://doi.org/10.3390/molecules26092645.
[150] H. Zhang, W. Zhao, L. Liu, W. Wen, X. Jing, X. Wang, Switchable deep eutectic solvents for sustainable extraction of β-carotene from millet, Microchemical Journal 187 (2023) 108369, https://doi.org/10.1016/j.microc.2022.108369.
[151] H. Zhang, W. Zhao, T. Bai, L. Fu, Z. Chen, X. Jing, X. Wang, Sustainable extraction of polyphenols from millet using switchable deep eutectic solvents, LWT 170 (2022) 114082, https://doi.org/10.1016/j.lwt.2022.114082.
[152] T. El Achkar, H. Greige-Gerges, S. Fourmentin, Basics and properties of deep eutectic solvents: a review, Environmental Chemistry Letters 19 (2021) 3397–3408, https://doi.org/10.1007/s10311-021-01225-8.
[153] C. Huang, Y. Zhan, J. Cheng, J. Wang, X. Meng, X. Zhou, G. Fang, A.J. Ragauskas, Facilitating enzymatic hydrolysis with a novel guaiacol-based deep eutectic solvent pretreatment, Bioresource Technology 326 (2021) 124696, https://doi.org/10.1016/j.biortech.2021.124696.
[154] Y. Zhang, X. Zhang, Z. Zhang, Z. Chen, X. Jing, X. Wang, Effect of high hydrostatic pressure treatment on the structure and physicochemical properties of millet gliadin, LWT 154 (2022) 112755, https://doi.org/10.1016/j.lwt.2021.112755.
[155] T. Liqin, L. Haocheng, W. Jing, X. Yujuan, T. Wenni, L. Lu, Y. Yuanshan, L. Xian, F. Manqin, Study on ultrahigh-pressure extraction technology on properties of yellow extract from gardenia fruit, Journal of Food Composition and Analysis 104 (2021) 104186, https://doi.org/10.1016/j.jfca.2021.104186.
[156] Y. Guo, H. Wang, Z. Chen, X. Jing, X. Wang, Determination of methomyl in grain using deep eutectic solvent-based extraction combined with fluorescence-based enzyme inhibition assays, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 266 (2022) 120412, https://doi.org/10.1016/j.saa.2021.120412.
[157] J. Zeng, Y. Dou, N. Yan, N. Li, H. Zhang, J.-N. Tan, Optimizing ultrasound-assisted deep eutectic solvent extraction of bioactive compounds from Chinese wild rice, Molecules 24 (2019) 2718, https://doi.org/10.3390/molecules24152718.
[158] J. Dong, L. Wang, J. Lü, Y. Zhu, R. Shen, Structural, antioxidant and adsorption properties of dietary fiber from foxtail millet (*Setaria italica*) bran, Journal of the Science of Food and Agriculture 99 (2019) 3886–3894, https://doi.org/10.1002/jsfa.9611.
[159] A.C. De Aguiar, J. Viganó, A.G. da Silva Anthero, A.L.B. Dias, M.D. Hubinger, J. Martínez, Supercritical fluids and fluid mixtures to obtain high-value compounds from Capsicum peppers, Food Chemistry: X 13 (2022) 100228, https://doi.org/10.1016/j.fochx.2022.100228.
[160] T. Ahmad, F.A. Masoodi, S.A. Rather, S.M. Wani, A. Gull, Supercritical fluid extraction: a review, Journal of Biological and Chemical Chronicles 5 (2019) 114–122, https://doi.org/10.33980/jbcc.2019.v05i01.019.
[161] P.A. Uwineza, A. Waśkiewicz, Recent advances in supercritical fluid extraction of natural bioactive compounds from natural plant materials, Molecules 25 (2020) 3847, https://doi.org/10.3390/molecules25173847.
[162] S. Qamar, Y.J. Manrique, H.S. Parekh, J.R. Falconer, Development and optimization of supercritical fluid extraction setup leading to quantification of 11 cannabinoids derived from medicinal cannabis, Biology 10 (2021) 481, https://doi.org/10.3390/biology10060481.

[163] H. Ahangari, J.W. King, A. Ehsani, M. Yousefi, Supercritical fluid extraction of seed oils – a short review of current trends, Trends in Food Science & Technology 111 (2021) 249–260, https://doi.org/10.1016/j.tifs.2021.02.066.
[164] N.U. Sruthi, P.S. Rao, B.D. Rao, Decortication induced changes in the physico-chemical, anti-nutrient, and functional properties of sorghum, Journal of Food Composition and Analysis 102 (2021) 104031, https://doi.org/10.1016/j.jfca.2021.104031.
[165] Y. Xiong, P. Zhang, R.D. Warner, Z. Fang, Sorghum grain: from genotype, nutrition, and phenolic profile to its health benefits and food applications, Comprehensive Reviews in Food Science and Food Safety 18 (2019) 2025–2046, https://doi.org/10.1111/1541-4337.12506.
[166] A. Ubeyitogullari, S.S.H. Rizvi, Production of high-purity phospholipid concentrate from buttermilk powder using ethanol-modified supercritical carbon dioxide, Journal of Dairy Science 103 (2020) 8796–8807, https://doi.org/10.3168/jds.2020-18697.
[167] P. Tyagi, K.S. Salem, M.A. Hubbe, L. Pal, Advances in barrier coatings and film technologies for achieving sustainable packaging of food products – a review, Trends in Food Science & Technology 115 (2021) 461–485, https://doi.org/10.1016/j.tifs.2021.06.036.
[168] G. Cottone, S. Giuffrida, S. Bettati, S. Bruno, B. Campanini, M. Marchetti, S. Abbruzzetti, C. Viappiani, A. Cupane, A. Mozzarelli, L. Ronda, More than a confinement: "soft" and "hard" enzyme entrapment modulates biological catalyst function, Catalysts 9 (2019) 1024, https://doi.org/10.3390/catal9121024.
[169] M. Kieliszek, K. Pobiega, K. Piwowarek, A.M. Kot, Characteristics of the proteolytic enzymes produced by lactic acid bacteria, Molecules 26 (2021) 1858, https://doi.org/10.3390/molecules26071858.
[170] U. Ejaz, M. Sohail, A. Ghanemi, Cellulases: from bioactivity to a variety of industrial applications, Biomimetics 6 (2021) 44, https://doi.org/10.3390/biomimetics6030044.
[171] C. Holland, P. Ryden, C.H. Edwards, M.M.-L. Grundy, Plant cell walls: impact on nutrient bioaccessibility and digestibility, Foods 9 (2020) 201, https://doi.org/10.3390/foods9020201.
[172] S.J. Marathe, S.B. Jadhav, S.B. Bankar, K. Kumari Dubey, R.S. Singhal, Improvements in the extraction of bioactive compounds by enzymes, Current Opinion in Food Science 25 (2019) 62–72, https://doi.org/10.1016/j.cofs.2019.02.009.
[173] S.A. Kulkarni, J.M. Sharma, A. Bobade, Antimicrobial power of endophytes from medicinal plants and enzyme production, International Journal of Creative Research Thoughts 9 (2021) 173–181.
[174] C.A. Nsude, T.C. Ezike, A.L. Ezugwu, O.E. Eje, I.N.E. Onwurah, F.C. Chilaka, Kinetics and thermodynamic properties of pectinase obtained from trichoderma longibrachiatum MT321074, Tropical Journal of Natural Product Research 6 (2022) 2063–2072, https://doi.org/10.26538/tjnpr/v6i12.28.
[175] N. Chiranthika, A. Chandrasekara, K. Gunathilake, In-vitro α-amylase and amyloglucosidase inhibitory activities of selected underutilized cereals, yams and root crops, Journal of Medicinal Plants Studies. 9 (2021) 105–110, https://doi.org/10.22271/plants.2021.v9.i3b.1271.
[176] M.A. Farooq, S. Ali, A. Hassan, H.M. Tahir, S. Mumtaz, S. Mumtaz, Biosynthesis and industrial applications of $α$-amylase: a review, Archives of Microbiology 203 (2021) 1281–1292, https://doi.org/10.1007/s00203-020-02128-y.
[177] M. Karaś, A. Jakubczyk, U. Szymanowska, K. Jędrka, S. Lewicki, U. Złotek, Different temperature treatments of millet grains affect the biological activity of protein hydrolyzates and peptide fractions, Nutrients 11 (2019) 550, https://doi.org/10.3390/nu11030550.
[178] M. Kazami, Y. Tsujii, M. Uchino, E. Sakaguchi, K. Takano, The rice grain localization of endosperm enzyme activity, Food Science and Technology Research 23 (2017) 151–155, https://doi.org/10.3136/fstr.23.151.
[179] M. Akbarian, A. Khani, S. Eghbalpour, V.N. Uversky, Bioactive peptides: synthesis, sources, applications, and proposed mechanisms of action, International Journal of Molecular Sciences 23 (2022) 1445, https://doi.org/10.3390/ijms23031445.
[180] C.M. Ajila, S.K. Brar, M. Verma, R.D. Tyagi, S. Godbout, J.R. Valéro, Extraction and analysis of polyphenols: recent trends, Critical Reviews in Biotechnology 31 (2011) 227–249, https://doi.org/10.3109/07388551.2010.513677.
[181] P.B. Devi, R. Vijayabharathi, S. Sathyabama, N.G. Malleshi, V.B. Priyadarisini, Health benefits of finger millet (Eleusine coracana L.) polyphenols and dietary fiber: a review, Journal of Food Science and Technology 51 (2014) 1021–1040, https://doi.org/10.1007/s13197-011-0584-9.
[182] W. Zhang, G. Zeng, Y. Pan, W. Chen, W. Huang, H. Chen, Y. Li, Properties of soluble dietary fiber-polysaccharide from papaya peel obtained through alkaline or ultrasound-assisted alkaline extraction, Carbohydrate Polymers 172 (2017) 102–112, https://doi.org/10.1016/j.carbpol.2017.05.030.
[183] C. Chen, L.-J. You, A.M. Abbasi, X. Fu, R.H. Liu, Optimization for ultrasound extraction of polysaccharides from mulberry fruits with antioxidant and hyperglycemic activity in vitro, Carbohydrate Polymers 130 (2015) 122–132, https://doi.org/10.1016/j.carbpol.2015.05.003.
[184] C. Sun, L. Qu, L. Wu, X. Wu, R. Sun, Y. Li, Advances in analysis of nitrated polycyclic aromatic hydrocarbons in various matrices, TrAC Trends in Analytical Chemistry 127 (2020) 115878, https://doi.org/10.1016/j.trac.2020.115878.
[185] D. Luthria, D. Vinjamoori, K. Noel, J. Ezzell, Accelerated solvent extraction, in: Oil Extraction and Analysis, AOCS Publishing, Urbana, IL, USA, 2019, pp. 25–38. https://books.google.co.in/books?hl=en&lr=&id=aCGeDwAAQBAJ&oi=fnd&pg=PA25&dq=Accelerated+solvent+extraction&ots=c2GtpA_zsY&sig=CLVLf-t26X_IgMykgF-vN9KJ4hQ&redir_esc=y#v=onepage&q=Accelerated.
[186] S. Raghunath, S. Budaraju, S.M.T. Gharibzahedi, M. Koubaa, S. Roohinejad, K. Mallikarjunan, Processing technologies for the extraction of value-added bioactive compounds from tea, Food Engineering Reviews 15 (2023) 276–308, https://doi.org/10.1007/s12393-023-09338-2.
[187] G. Alvarez-Rivera, M. Bueno, D. Ballesteros-Vivas, J.A. Mendiola, E. Ibañez, Pressurized liquid extraction, in: Liquid-Phase Extraction, Elsevier, 2020, pp. 375–398, https://doi.org/10.1016/B978-0-12-816911-7.00013-X.
[188] T. Tolcha, T. Gemechu, S. Al-Hamimi, N. Megersa, C. Turner, Multivariate optimization of a combined static and dynamic supercritical fluid extraction method for trace analysis of pesticides pollutants in organic honey, Journal of Separation Science 44 (2021) 1716–1726, https://doi.org/10.1002/jssc.202100047.
[189] S. Zia, M.R. Khan, M.A. Shabbir, A. Aslam Maan, M.K.I. Khan, M. Nadeem, A.A. Khalil, A. Din, R.M. Aadil, An inclusive overview of advanced thermal and nonthermal extraction techniques for bioactive compounds in food and food-related matrices, Food Reviews International 38 (2022) 1166–1196, https://doi.org/10.1080/87559129.2020.1772283.
[190] P.H. Santos, J.C. Kammers, A.P. Silva, J.V. Oliveira, H. Hense, Antioxidant and antibacterial compounds from feijoa leaf extracts obtained by pressurized liquid extraction and supercritical fluid extraction, Food Chemistry 344 (2021) 128620, https://doi.org/10.1016/j.foodchem.2020.128620.
[191] W. Setyaningsih, I.E. Saputro, M. Palma, C.G. Barroso, Pressurized liquid extraction of phenolic compounds from rice (Oryza sativa) grains, Food Chemistry 192 (2016) 452–459, https://doi.org/10.1016/j.foodchem.2015.06.102.
[192] S. Sirichokworrakit, H. Rimkeeree, W. Chantrapornchai, U. Sukatta, Comparative study on conventional, accelerated solvent extraction and ultrasonic-assisted extraction of total phenolic and anthocyanin contents and antioxidant activities from Riceberry bran, Agriculture and Natural Resources 56 (2022) 243–254, https://doi.org/10.34044/j.anres.2022.56.2.03.
[193] R.L.J. Almeida, N.C. Santos, C.E.S. Muniz, R. da Silva Eduardo, R. de Almeida Silva, C.A.C. Ribeiro, G.A. da Costa, M.J. de Figueiredo, P.O. Galdino, E.S. dos Santos, Red rice starch modification - combination of the non-thermal method with a pulsed electric field (PEF) and enzymatic method using α-amylase, International Journal of Biological Macromolecules 253 (2023) 127030, https://doi.org/10.1016/j.ijbiomac.2023.127030.
[194] R.N. Arshad, Z. Abdul-Malek, U. Roobab, M.A. Munir, A. Naderipour, M.I. Qureshi, A. El-Din Bekhit, Z.-W. Liu, R.M. Aadil, Pulsed electric field: a potential alternative towards a sustainable food processing, Trends in Food Science & Technology 111 (2021) 43–54, https://doi.org/10.1016/j.tifs.2021.02.041.
[195] E. Demir, S. Tappi, K. Dymek, P. Rocculi, F. Gómez Galindo, Reversible electroporation caused by pulsed electric field – opportunities and challenges for the food sector, Trends in Food Science {\&} Technology 139 (2023) 104120, https://doi.org/10.1016/j.tifs.2023.104120.
[196] M.M.A.N. Ranjha, R. Kanwal, B. Shafique, R.N. Arshad, S. Irfan, M. Kieliszek, P.Ł. Kowalczewski, M. Irfan, M.Z. Khalid, U. Roobab, R.M. Aadil, A critical review on pulsed electric field: a novel technology for the extraction of phytoconstituents, Molecules 26 (2021) 4893, https://doi.org/10.3390/molecules26164893.
[197] E. Puértolas, M. Koubaa, F.J. Barba, An overview of the impact of electrotechnologies for the recovery of oil and high-value compounds from vegetable oil industry: energy and economic cost implications, Food Research International 80 (2016) 19–26, https://doi.org/10.1016/j.foodres.2015.12.009.
[198] A. Ricci, G. Parpinello, A. Versari, Recent advances and applications of pulsed electric fields (PEF) to improve polyphenol extraction and color release during red winemaking, Beverages 4 (2018) 1–18, https://doi.org/10.3390/beverages4010018.
[199] J. Xi, Z. Li, Y. Fan, Recent advances in continuous extraction of bioactive ingredients from food-processing wastes by pulsed electric fields, Critical Reviews in Food Science and Nutrition 61 (2021) 1738–1750, https://doi.org/10.1080/10408398.2020.1765308.

[200] U.C. Lohani, K. Muthukumarappan, Application of the pulsed electric field to release bound phenolics in sorghum flour and apple pomace, Innovative Food Science & Emerging Technologies 35 (2016) 29–35, https://doi.org/10.1016/j.ifset.2016.03.012.
[201] S. Teh, B.E. Niven, A.E.A. Bekhit, A. Carne, E.J. Birch, Microwave and pulsed electric field assisted extractions of polyphenols from defatted canola seed cake, International Journal of Food Science & Technology 50 (2015) 1109–1115, https://doi.org/10.1111/ijfs.12749.
[202] A. Vallverdú-Queralt, G. Oms-Oliu, I. Odriozola-Serrano, O. Lamuela-Raventós Rosa Ma, Martín-Belloso, P. Elez-Martínez, Metabolite profiling of phenolic and carotenoid contents in tomatoes after moderate-intensity pulsed electric field treatments, Food Chemistry 136 (2013) 199–205, https://doi.org/10.1016/j.foodchem.2012.07.108.
[203] T. Gachovska, D. Cassada, J. Subbiah, M. Hanna, H. Thippareddi, D. Snow, Enhanced anthocyanin extraction from red cabbage using pulsed electric field processing, Journal of Food Science 75 (2010) E323–E329, https://doi.org/10.1111/j.1750-3841.2010.01699.x.
[204] N. Salee, S. Naruenartwongsakul, W. Chaiyana, A. Yawootti, K. Hunsakul, B. Tinpovong, N. Utama-Ang, Comparison of pulse electric field, microwave and ultrasonic pretreatment prior to black rice extraction on antioxidant and sirtuin1 enzyme stimulating activities, Food Science and Technology 43 (2023) e123022, https://doi.org/10.1590/fst.123022.
[205] N. Salee, W. Chaiyana, A. Yawootti, S. Naruenartwongsakul, W. Klangpetch, P. Walter, N. Utama-ang, Optimization of the pulse electric field assisted extraction of black rice grain for antioxidant and sirtuin1 enzyme stimulation activities, Scientific Reports 12 (2022) 6459, https://doi.org/10.1038/s41598-022-10272-2.
[206] S. Rajchasom, P. Sombatnan, C. Kantala, J. Tancharoenrat Vuttijamnong, Effect of pulse electric field assisted extraction on anthocyanin content and antioxidant activity of purple rice, SNRU Journal of Science and Technology 14 (2022) 245162, https://doi.org/10.55674/snrujst.v14i2.245162.
[207] V. Quagliariello, R.V. Iaffaioli, M. Falcone, G. Ferrari, G. Pataro, F. Donsì, Effect of pulsed electric fields – assisted extraction on anti-inflammatory and cytotoxic activity of brown rice bioactive compounds, Food Research International 87 (2016) 115–124, https://doi.org/10.1016/j.foodres.2016.07.005.
[208] S. Ashraf, R. Scholor, M. Sood, A. Professor, J.D. Bandral, A. Professor, M. Trilokia, J. Kashmir, I.M. Manzoor, M. Manzoor, Food irradiation: a review, International Journal of Chemical Studies 7 (2019) 131–136. https://www.researchgate.net/publication/332548321.
[209] D.K. Aswal, S. V Nakhe, P. Shukla, N. Chaudhary, T. Ganguli, B.N. Upadhyay, An overview of key enabling technologies for DAE's nuclear programme, CURRENT SCIENCE 123 (2022) 353.
[210] X. Fei, W. Jia, J. Wang, T. Chen, Y. Ling, Study on enzymatic hydrolysis efficiency and physicochemical properties of cellulose and lignocellulose after pretreatment with electron beam irradiation, International Journal of Biological Macromolecules 145 (2020) 733–739, https://doi.org/10.1016/j.ijbiomac.2019.12.232.
[211] R.U. Erdemir, G. Kilic, D. Sen Baykal, G. Almisned, S.A.M. Issa, H.M.H. Zakaly, A. Ene, H.O. Tekin, Diagnostic and therapeutic radioisotopes in nuclear medicine: determination of gamma-ray transmission factors and safety competencies of high-dense and transparent glassy shields, Open Chemistry 20 (2022) 517–524, https://doi.org/10.1515/chem-2022-0167.
[212] L.K. Mohamed, M.A. Sulieman, A.E.A. Yagoub, M.A. Mohammed, H.F. Alhuthayli, I.A. Mohamed Ahmed, S.A. Almaiman, M.A. Alfawaz, M.A. Osman, A. B. Hassan, Changes in phytochemical compounds and antioxidant activity of two irradiated sorghum (sorghum bicolor (L.) monech) cultivars during the fermentation and cooking of traditional Sudanese Asida, Fermentation 8 (2022) 60, https://doi.org/10.3390/fermentation8020060.
[213] A.B. Hassan, S.A. Al Maiman, M.A. Mohammed, G.M. Alshammari, D.A. Alkhudhayri, H.F. Alhuthayli, M.A. Alfawaz, M.A. Osman, Effect of natural fermentation on the chemical composition, mineral content, phytochemical compounds, and antioxidant activity of ziziphus spina-christi (L.) "nabag" seeds, Processes 9 (2021) 1228, https://doi.org/10.3390/pr9071228.
[214] L. Fan, W. Fan, Y. Mei, L. Liu, L. Li, Z. Wang, L. Yang, Mechanochemical assisted extraction as a green approach in preparation of bioactive components extraction from natural products - a review, Trends in Food Science & Technology 129 (2022) 98–110, https://doi.org/10.1016/j.tifs.2022.09.009.
[215] L. Zheng, X. Zhu, J. Wang, W. Su, Mechanochemical-assisted extraction and enzymatic hydrolysis of calcium phytate from defatted rice bran, Chemical and Biological Technologies in Agriculture 10 (2023) 53, https://doi.org/10.1186/s40538-023-00424-5.
[216] K. Wu, T. Ju, Y. Deng, J. Xi, Mechanochemical assisted extraction: a novel, efficient, eco-friendly technology, Trends in Food Science & Technology 66 (2017) 166–175, https://doi.org/10.1016/j.tifs.2017.06.011.
[217] S. Wang, R. Zhang, X. Song, M. Wei, T. Xie, J. Cao, Mechanochemical-assisted extraction of active alkaloids from plant with solid acids, ACS Sustainable Chemistry & Engineering 7 (2019) 197–207, https://doi.org/10.1021/acssuschemeng.8b02902.