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Review

Microwave-assisted extraction with natural deep eutectic solvents for polyphenol recovery from agrifood waste: Mature for scaling-up?

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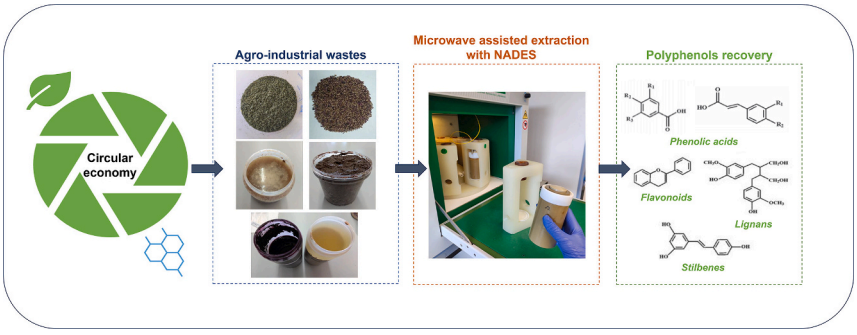
HIGHLIGHTS

- Agrifood waste is a great source of valuable phenolic compounds.
- Polyphenols are beneficial for human health due to antioxidant features.
- The extraction process is a key point for an efficient recovery of polyphenols.
- Microwave-assisted extraction shows a great performance to recover polyphenols.
- Natural deep eutectic solvents are a green alternative to conventional solvents.

GRAPHICAL ABSTRACT



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ABSTRACT

Agrifood industries generate large amounts of waste that may result in remarkable environmental problems, such as soil and water contamination. Therefore, proper waste management and treatment have become an environmental, economic, and social challenge. Most of these wastes are exceptionally rich in bioactive compounds (e.g., polyphenols) with potential applications in the food, cosmetic, and pharmaceutical industries. Indeed, the recovery of polyphenols from agrifood waste is an example of circular bioeconomy, which contributes to the valorization of waste while providing solutions to environmental problems. In this context, unconventional extraction techniques at the industrial scale, such as microwave-assisted extraction (MAE), which has demonstrated its efficacy at the laboratory level for analytical purposes, have been suggested to search for more efficient recovery procedures. On the other hand, natural deep eutectic solvents (NADES) have been proposed as an efficient and green alternative to typical extraction solvents. This review aims to provide comprehensive insights regarding the extraction of phenolic compounds from agrifood waste. Specifically, it focuses on the utilization of MAE in conjunction with NADES. Moreover, this review delves into the possibilities of recycling and reusing NADES for a more sustainable and cost-efficient industrial application. The results obtained with the MAE-

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NADES approach show its high extraction efficiency while contributing to green practices in the field of natural product extraction. However, further research is necessary to improve our understanding of these extraction strategies, optimize product yields, and reduce overall costs, to facilitate the scaling-up.

1. Introduction

Agrifood industries generate large amounts of solid and liquid waste, which represent a noticeable environmental problem. These residues/by-products may be harmful to the environment but are exceptionally rich in bioactive compounds, such as polyphenols, proteins, vitamins, carotenoids, and alkaloids (Mir-Cerdà et al., 2023a), with potential applications in the food, cosmetic, and pharmaceutical industries (Tapia-Quirós et al., 2022a; Tapia-Quirós et al., 2020). Therefore, the recovery of phytochemicals from these residues holds significant importance for agrifood industries within the context of sustainability and the circular economy.

Polyphenols are plant metabolites with more than one phenol group in their structure. They are beneficial for human health due to their high antioxidant character, neutralizing the formation of free radicals involved in oxidation processes. Several studies have correlated the intake of these compounds to positive effects on diseases such as cancer, diabetes, and hypertension, among others (Tapia-Quirós et al., 2022b).

Organic solvents are commonly used in extraction processes (e.g., methanol, acetone, or ethyl acetate) but present some disadvantages, such as toxicity, low vapor pressures, flammability, or pollution, among others (Isci and Kaltschmitt, 2022). When considering the application of recovered phenolic compounds in the food, pharmaceutical, or cosmetic industries, ethanol and water are the most compatible options (Tapia-Quirós et al., 2022a). The exploration of alternative green solvents has resulted in the development of natural deep eutectic solvents (NADES) (Isci and Kaltschmitt, 2022), which consist of a mixture of a hydrogen bond acceptor (e.g., choline chloride) and a hydrogen bond donor (e.g., glycerol) to formulate eutectic mixtures. NADES have been suggested to recover phenolic compounds as a green alternative to conventional organic solvents, offering enhanced extraction efficiency and quality of the extracts (Chanioti et al., 2021).

Using NADES as extractant, the recovery of phenolic compounds from agrifood residues can be achieved by different extraction processes (Chanioti et al., 2021). Conventional solid-liquid extraction (SLE) relies on the contact of the sample with appropriate solvents for a certain time. Shaking and heating speed up and/or enhance the extraction of bioactive compounds (Tapia-Quirós et al., 2022a). Some authors use the term homogenization-assisted extraction (HAE) to refer the SLE with high-speed mechanical shearing, mixing, fluid cutting action, and smashing without the use of pressure (Chanioti et al., 2021; Chanioti and Tzia, 2018). Ultrasound-assisted extraction (UAE) consists of the application of high-frequency waves ($\geq$2 MHz), which produce negative pressure, change the physicochemical properties of the medium, allow the formation of cavitation bubbles, and, therefore, break the cell membranes in the sample promoting contact between solvent and target compounds (Tapia-Quirós et al., 2020; Tapia-Quirós et al., 2022b). High hydrostatic pressure-assisted extraction (HHPAE) operates under very high pressures, ranging from 100 to 1000 MPa, which facilitates the penetration of the solvent into the solid matrix releasing the compounds of interest (Chanioti et al., 2021; Chanioti and Tzia, 2018). Microwave-assisted extraction (MAE), based on the direct impact of microwave radiation, is the focus of this review. The microwave energy heats the polar solvents in contact with the solid samples, increasing the internal pressure and promoting cell wall rupture, which releases bioactive compounds to the solvent (Tapia-Quirós et al., 2020; Tapia-Quirós et al., 2022b).

After the extraction step, obtaining information about the phenolic compositional characteristics of the extract can be achieved through two main approaches as follows: (i) chromatographic techniques, which provide a phenolic profile and allow the quantification of individual polyphenols; (ii) spectrophotometric assays, which estimate the total polyphenol content or the antioxidant capacity (Tapia-Quirós et al., 2022b). The most frequently used chromatographic techniques for polyphenols analysis are high-performance liquid chromatography (HPLC) or ultra-high-performance liquid chromatography (UHPLC) with spectroscopic ultraviolet-visible (UV–vis) and/or fluorescence (FLD) detection, or coupled with mass spectrometry (MS) (Mir-Cerdà et al., 2023a; Tapia-Quirós et al., 2022b). Regarding spectrophotometric assays, the Folin-Ciocalteu (FC) assay is commonly used to estimate the total polyphenol content in vegetal products. Also, the antioxidant capacity can be measured by different methods, such as ferric reducing antioxidant power (FRAP), cupric reducing antioxidant capacity (CUPRAC) assay, 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay, 2,20-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) assay, and oxygen radical absorbance capacity (ORAC) (Tapia-Quirós et al., 2022b).

Following the compositional characterization of the extracts, another important step is their purification to generate products focused on specific applications. In some cases, a concentrate of phenolic compounds may be of interest which seeks to maximize the antioxidant or antiradical capacity without paying attention to any particular compound. Another possibility pursues the fractionation of specific families of compounds or isolation of individual compounds (in general, the most abundant and remarkable).

In this manuscript, the studies on the use of NADES combined with MAE for the extraction of phenolic compounds from the most relevant agrifood waste matrices are reviewed below, including those related to olive oil, winemaking, brewing, fruit and vegetable processing, and medicinal plants. Another section deals with the purification of the obtained extracts and the recycling and reuse of NADES.

2. Extraction of phenolic compounds from agrifood waste

This section reports studies about the extraction of polyphenols from different agrifood waste focusing on the use of NADES. The majority of NADES formulations used for polyphenols are composed of choline chloride as proton acceptor. In terms of proton donors, a diversity of compounds has been assessed, such as glycerol, lactic acid, urea, glucose, etc. (Fig. 1). Water is another common component of NADES

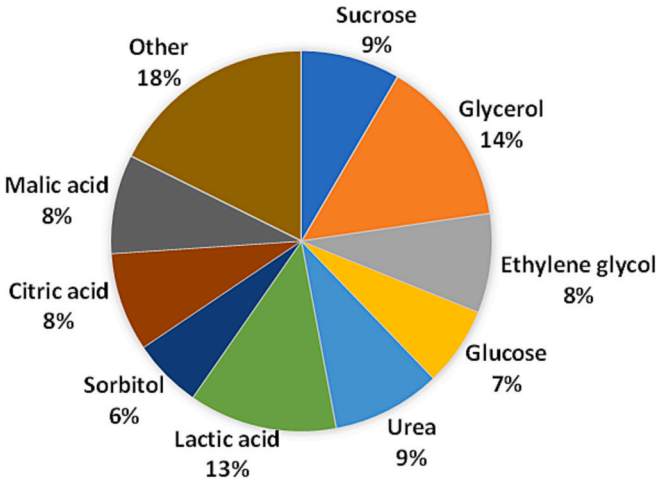

Fig. 1. Main hydrogen bond donor (HBD) of NADES mixtures for polyphenol recovery by MAE.

mixtures, with percentages generally between 10 and 30 %. Ternary mixtures containing two different donors have also been proposed. With regard to the technique, the extraction of polyphenols using NADES has been investigated with various processing techniques, such as SLE, MAE, or UAE (Fig. 2).

Table 1 summarizes the main features of representative works on the application of NADES to the extraction of polyphenolic compounds from agrifood waste by MAE and other extractive techniques. The number of recent studies is already considerable, and we detect that this approach is increasing because of its indisputable benefits (Fig. 3). However, it is worth mentioning that most of the studies carried out so far have been limited to a laboratory scale.

Based on the content of Table 1, some general considerations are as follows. First, it is proved that NADES is a good alternative to conventional solvents to recover phenolic compounds in the waste matrices. Extractive yields, both in overall antioxidant activity and in specific compounds, are commonly higher. Table S1 (Supplementary material) shows the reported NADES systems to extract specific polyphenols. It can be observed that systems based on choline:glycerol or choline:propylene glycol extract polyphenols with a wide range of physicochemical properties, from phenolic acids to highly complex flavonoids. However, with the currently available information, it is challenging to provide guidelines to achieve a selective extraction of polyphenols, let alone a specific type of polyphenol or a given individual compound. At present, there is no criterion to find out, a priori, which mixture may be more convenient for a given case. It seems, otherwise, that it is an empirical issue in which a trial-and-error procedure will lead to establishing the most recommendable conditions. More extensive considerations on this topic are included in Section 2.8.

Concerning the extractive technique, MAE is a good alternative as has been demonstrated in most of the studies that compare several extraction techniques. In general, it provides comparable benefits in terms of extraction performance, while processing times are usually shorter than those of other techniques, such as UAE. However, power applied to MAE should be carefully optimized to avoid or minimize the degradation of the most labile compounds when subjected to high temperatures within the cells of the microwave system. On the other hand, MAE is considered a perfectly scalable technique to be implemented at the pilot or industrial plant level. Nevertheless, it is important to note that there is a scarcity of studies specifically addressing the scalability of the MAE-NADES approach. To emphasize the efforts in this sense, we have included Table 2 to summarize the principal scaling attempts and expanded comments in the Section 2.7. To date, the systems developed at the pilot plant level have made it possible to process quantities of waste ranging from several dozen grams to several kilos. However, our bibliographic search has not found any publication in which the MAE technology is applied on an industrial scale to revalorize agri-food waste.

The main waste types or matrices explored for the recovery of phenolic compounds, structured by typology, are discussed below.

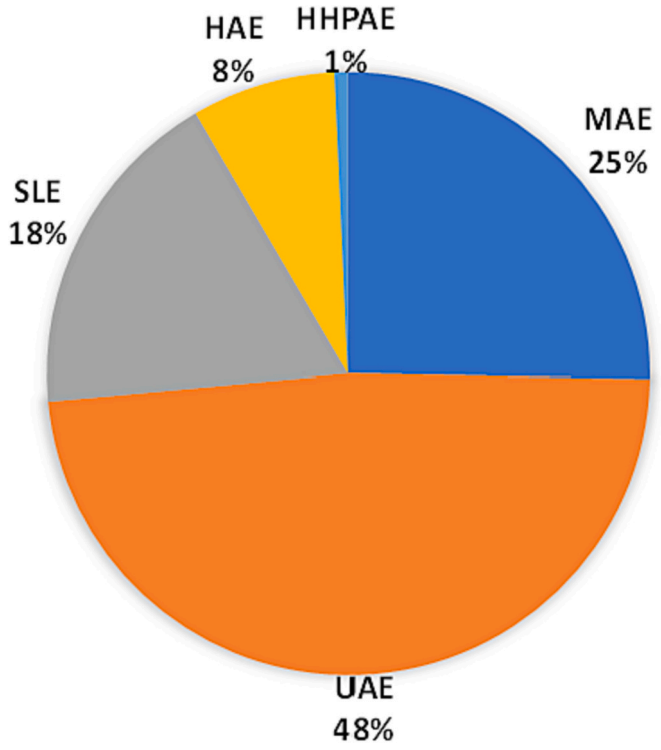

Fig. 2. Main extraction techniques for polyphenols recovery using NADES. MAE, microwave-assisted extraction; UAE, ultrasound-assisted extraction; SLE, solid-liquid extraction (by mechanical stirring and heating); HAE, homogenization-assisted extraction; HHPAE, high hydrostatic pressure-assisted extraction.

2.1. Olive mill by-products

Olive oil production is one of the major economic activities in Europe with 98 % of the world production of 2.55 million metric tons per year (Tapia-Quirós et al., 2020; Tapia-Quirós et al., 2022c; Dermeche et al., 2013). Olive oil production generates about 10 million tons of waste per year, which includes solid and liquid waste, with olive pomace, olive leaves, and olive mill wastewater being the main ones (Tapia-Quirós et al., 2022a; Tapia-Quirós et al., 2022b). Olive pomace production is higher than 2.8 million tons/year worldwide (Nunes et al., 2016; Ravindran and Jaiswal, 2016); olive mill wastewater production is between 7 and >30 million m^3 per year worldwide (Dermeche et al., 2013); and olive leaves account for up to 10 % of the total weight of the olives at olive-oil mills (Alañón et al., 2020). The principal phenolic compounds found in these residues are 3-hydroxytyrosol, oleuropein, and flavonoids such as rutin, and luteolin glycosides. Some illustrative publications on the extraction of these phytochemicals from this variety of olive waste matrices are commented as follows.

Chanioti and Tzia (2018) studied the extraction of phenolic compounds from olive pomace using different NADES (choline chloride with citric acid, lactic acid, maltose, and glycerol) and innovative extraction techniques, including HAE, MAE, UAE, and HHPAE. The authors also compared NADES with conventional solvents such as ethanol and water, demonstrating that the extracts obtained using MAE and choline chloride with lactic acid or glycerol as the solvents exhibited comparable properties in terms of total phenolic content, radical scavenging activity, and oleuropein content when compared to the ethanol extracts obtained using HAE and MAE. These results align with findings of MAE studies that show that NADES are rapidly heated, reducing their viscosity and surface tension and leading to an enhanced extraction process with improved extraction yield (Chanioti and Tzia, 2018; Yao et al., 2015). Chanioti et al. (2021) also found that choline chloride:lactic acid (1:2) solvent achieved excellent extraction yields in terms of total phenolic content —29.57 mg gallic acid equivalents (GAE)/g dry weight (dw)— and antioxidant activity —17.51 g Trolox equivalents (TE)/g dw DPPH— compared to ethanol:water (70:30, v/v) when MAE was used as extraction technique. Thus, it was concluded that NADES show excellent absorption of microwave energy, making them suitable green solvents for MAE processes.

Other authors have also recommended MAE with NADES for the extraction of polyphenols from olive mill residues. For instance, Bonacci et al. (2020) studied the recovery of polyphenols from olive leaves and ripened olive drupes by MAE, and compared the performance of several NADES combinations (e.g., choline chloride with urea, glycerol, lactic acid, ethylene glycol, and citric acid, with 20 % of water) as well as water as extraction solvents. Choline chloride:glycerol (1:1), was the most effective solvent under the extraction conditions of 2:8 w/v with

Table 1
Application of combined MAE and NADES processing for the extraction of polyphenols from agrifood waste.

Food sector	Waste	Extraction technique	Evaluated solvents	Selected extraction conditions	Polyphenol concentration	Reference
Olive oil	Olive pomace	HAE, MAE, UAE, HHPAE	Ethanol:water (70:30, v/v), methanol, ChCl with caffeic acid, and LA (1:2)	HAE, ChCl:caffeic acid (1:2), 60 °C, 12,000 rpm	TPC: 34.08 mg GAE g^{-1} dw by FC	(Chanioti et al., 2021)
	Olive pomace	HAE, MAE, UAE, HHPAE	Ethanol:water (70:30, v/v), water, ChCl with CA, LA, maltose, and Gly (1:2), with 20 % of water	HAE, ChCl:CA (1:2) with 20 % of water, 60 °C, 12,000 rpm, 30 min	TPC: 34.08 mg GAE g^{-1} dw by FC	(Chanioti and Tzia, 2018)
	Olive leaves	MAE	Methanol:water (80:20 v/v), ChCl with LA, oxalic acid, tartaric acid, 1,4-butanediol, ethylene glycol, xylitol, 1,2-propanediol, maltose, and urea, (1:1–3:1) with water (0–70 %)	MAE, ChCl:ethylene glycol (1:2) with 43.3 % of water, 79.6 °C, 16.7 min	TPC: 28.52 mg GAE g^{-1} by FC; Oleuropein: 10.58 mg g^{-1} by HPLC-DAD-ESI-TOF-MS	(Alañón et al., 2020)
	Olive leaves, ripened olive drupes	MAE	Water, ChCl with urea, Gly, LA, ethylene glycol, and CA, (1:1–1:2) with 20 % of water	MAE, ChCl:Gly (1:1) with 20 % of water, 2:8 w/v, 80 °C, 800 W, 10 and 30 min	Olive leaves: 416.08 mg oleuropein L^{-1} by HPLC; Ripe olive extracts: 88287.57 mg oleuropein L^{-1} by HPLC	(Bonacci et al., 2020)
	Olive leaves	SLE, MAE	ChCl with urea or acetic acid (1:2)	MAE, ChCl:acetic acid (1:2) with ethanol (80:20 w/w), 1:10 w/v, 70 °C, 500 W, 5 and 30 min	TPC: 4438 mg GAE L^{-1} by FC	(Boli et al., 2022)
Wine	Grape skin	SLE, UAE, MAE	Water, methanol:water (70:30, v/v), methanol:water:HCl (70:29:1, v/v/v), ChCl with Gly, oxalic acid, malic acid, sorbose, and proline:malic acid (1:1–1:2; 1:1:1) with water (10–50 %)	UAE, ChCl:oxalic acid (1:1) with 25 % of water, 65 °C, 50 min	Total free anthocyanins: ~30 mg g^{-1} dw by HPLC	(Cvjetko Bubalo et al., 2016)
Coffee	Spent coffee grounds	UAE, MAE	Ethanol:water (25–75 % ethanol); ChCl:Gly (1:3), ChCl:LA:water (1:2:2.5), ChCl:CA:water (1:1:4), betaine:CA:water (1:1:6), betaine: LA:water (1:2:2.5), betaine:Gly (1:3), with water (25–75 %)	MAE, betaine:Gly (1:3) with 50 % of water, 1 % solid-to-liquid ratio, 90 °C, 60 min	TPC: up to 30.9 mg GAE g^{-1} by FC; TFC: up to 22.8 mg CE g^{-1} by aluminum trichloride method	(Tzani et al., 2023)
	Spent coffee grounds	MAE	Ethanol:water 25:75 v/v, ChCl:Gly (0.5–1.5 M) with water (20–70 %)	MAE, ChCl:Gly (0.53 M ratio) with 20 % of water, 120 °C, 15 min	TPC: 0.48 mg GAE L^{-1} by FC	(López-Linares et al., 2021a)
Beer	Brewer's spent grain	MAE	Methanol:water (80:20 v/v); ChCl with ethylene glycol, LA, Gly, and 1,2-propanediol (1:2), with water (20–70 %)	MAE, ChCl:Gly (1:2) with 37.46 % of water, 100 °C, 13.30 min	TPC: 2.89 mg GAE L^{-1} by FC	(López-Linares et al., 2021b)
Fruits	Mulberry leaves	HRE, UAE, MAE	Ethanol, methanol, ChCl with CA, LA, malic acid, urea, sorbitol, ethylene glycol, 1,2-propanediol, Gly, 1,4-butanediol, sucrose, fructose, and glucose (1:2;1:1–2:1), with water (0–80 %)	MAE, ChCl:Gly (1:2) with 20 % of water, 1:20 w/v, 66 °C, 18 min	TPC: 8.352 mg g^{-1} by HPLC	(Gao et al., 2020)
	Chestnut shells	MAE	ChCl with malic acid, oxalic acid, CA, levulinic acid, and oxalic acid (1:1 or 1:2), with water (0–25 %)	MAE; ChCl:oxalic acid (1:1) with 13.5 % of water, 1:10 w/v, 85 °C, 60 min	TPC: 295.2 mg GAE g^{-1} dw by FC	(González-Rivera et al., 2021)
	Black jamun pulp	MAE	ChCl:CA (1:1) with water (10–40 %)	MAE, ChCl:CA (1:1) with 40 % of water, 48 mL g^{-1} liquid-solid ratio, 400 W, 4 min	TPC: 45 mg GAE g^{-1} by FC; TAC: 9.3 mg C3G g^{-1} by pH differential method	(Sharma and Dash, 2021)
	Fig leaves	UAE, MAE	Methanol, ChCl with galactose, proline, malic acid, xylitol, fructose, sucrose, CA, and glucose (1:1–5:2); Gly:xylitol:fructose (1:3:1–3:3:3) with water (10–20 %)	MAE; Gly:xylitol:fructose (3:3:3) with 20 % of water, 17.53 mL g^{-1} liquid-solid ratio, 64.46 °C, 24.43 min	Caffeoylmalic acid: 6.482 mg g^{-1} by HPLC; Psoralic acid-glucoside: 16.34 mg g^{-1} by HPLC; Rutin: 5.207 mg g^{-1} by HPLC; Psoralen: 15.22 mg g^{-1} by HPLC; Bergapten: 2.475 mg g^{-1} by HPLC	(Wang et al., 2017)
	Sour cherry pomace	HSE, UAE, MAE	Ethanol:water (50:50, v/v); 0.1 % HCl in MeOH; ChCl and malic acid, urea, and fructose (1:1), with 20 % of water	MAE, ChCl:malic acid (1:1) with 20 % of water, 40 °C, 90 W, 15 s	TPC: 3238.32 μg g^{-1} dw by HPLC	(Popovic et al., 2021)
	Ripe mango peel	HSE, SLE, MAE	ChCl with urea, sorbitol, sucrose, Gly, LA, and malic acid (1:1–3:1); sodium acetate with Gly, and LA (1:1–1:4), with water (0–100 %)	MAE, LA:sodium acetate: water (3:1:4), 59.82 mL g^{-1} liquid-solid ratio, 436.45 W, 19.66 min	TPC: 56.17 mg GAE g^{-1} dw by FC	(Pal and Jadeja, 2019b)
	Defatted date seeds	MAE	ChCl with formic acid, Gly, and urea (1:2) with water (5–15 %)	MAE, ChCl:formic acid (1:2), 1:20 w/v, 400 W, 50 s	TPC: 128 g GAE kg^{-1} by FC	(Kehili et al., 2022)
	Hazelnut pomace	MAE	ChCl with 1,2-butandiol, 1,2-propylene glycol, and Gly (1:4); ChCl:DL-malic acid:water (1:1:2); sucrose:LA:water (1:5:7); fructose: LA:water (1:5:5); sucrose:ChCl:water (1:4:4); fructose:ChCl:water (2:5:5); ethanol:water (80:20, v/v)	MAE, ChCl:1,2-propylene glycol (1:4) with 24 % of water, 18 mL 0.1 g^{-1} dw liquid-solid ratio, 92 °C, 38 min	Total antioxidant capacity: 0.17 to 0.24 mmol TE g^{-1} dw by CUPRAC	(Bener et al., 2022)

(*continued on next page*)

Table 1 (*continued*)

Food sector	Waste	Extraction technique	Evaluated solvents	Selected extraction conditions	Polyphenol concentration	Reference
	Pineapple peel	MAE	Water, ethanol, methanol, ethanol: water (50:50, v/v), methanol:water (50:50, v/v), ChCl:Gly (1:2) with 10 % of water, ChCl:malic acid (1:1.5) with 50 % of water	MAE, ChCl:Gly (1:2) with 10 % of water, 60.5 mL g^{-1} liquid-solid ratio, 67 °C drying temperature, 87 s	TPC: 7.98 mg GAE g^{-1} dw by FC	(Vargas-Serna et al., 2022)
	Raw mango peel	MAE	Water, methanol:water (70:30, v/v), ChCl with urea, sorbitol, malic acid, sucrose, Gly, and LA (1:1–3:1); sodium acetate with LA, and Gly (1:3), with water (0–100 %)	MAE, sodium acetate:Gly (1:3) with 20 % of water, 59.99 mL g^{-1} liquid-solid ratio, 440.18 W, 12.10 min	TPC: 155.28 mg GAE g^{-1} dw by FC	(Pal and Jadeja, 2022)
	Sour cherry peels	MAE	CA:ethylene glycol (1:4)	MAE, CA:ethylene glycol (1:4), 500 W, 180 s	TPC: 4.29–33.61 mg GAE g^{-1} fw by FC	(Kurtulbaş, 2022)
Vegetables	*Cajanus cajan* leaves	MAE	Ethanol:water (60:40, v/v), Ethanol: water (80:20, v/v), ChCl with Gly, 1,4-butanediol, ethylene glycol, glucose, sucrose, maltose, sorbitol, CA, malic acid, and LA; CA with glucose, and sucrose; LA with glucose, and sucrose, (1:2–1:4) with water (0–60 %)	MAE, ChCl:maltose (1:2) with 20 % of water, 1:30 w/v, 60 °C, 12 min	TPC: 30.63 mg g^{-1} by UPLC	(Wei et al., 2015)
	Onion peel	MAE, HSE, Soxhlet	ChCl with sucrose, urea, oxalic acid, and sorbitol (1:1–4:1) with water (4 mol)	MAE, ChCl:urea:water (1:2:4), 54.97 mL g^{-1} liquid-solid ratio, 100 W, 15.03 min	TPC: 80.45 mg GAE g^{-1} dw by FC	(Pal and Jadeja, 2019a)
	Spent onion skins	MAE	Temperature-responsive deep eutectic solvents (ethanolamine with o-cresol, m-cresol, and p-cresol)	Ethanolamine:m-cresol 1:1 (20 % water), 14 mg mL^{-1} solid-liquid ratio, 554 W, 76 °C, 16 min	Flavonoids: 47.83 mg g^{-1}	(Shang et al., 2022)
Plants	*Pyrola incarnata*	HSE, UAE, MAE	ChCl with ethyl glycol, Gly, 1,2-butanediol, 1,3-butanediol, 1,4-butanediol, 1,6-hexanediol, (1:1–1:6) with water (0–100 %)	MAE, ChCl:1,4-butanediol (1:4) with 30 % of water, 10 mL g^{-1} liquid-solid ratio, 70 °C, 20 min	Hyperin: 1.627 mg g^{-1}; 20-O-galloylhyperin: 4.958 mg g^{-1}; quercitrin: 0.041 mg g^{-1}; quercetin-O-rhamnoside: 0.089 mg g^{-1}; chimaphilin: 0.349 mg g^{-1}, by HPLC	(Yao et al., 2015)
	Eugenia uniflora	MAE	Malic acid with sorbitol (1:1), and glucose:fructose (1:1:1); LA:sucrose; ChCl with glucose, and LA, (1:1–1:3) with 20 % of water	MAE, ChCl:LA (1:3) with 20 % of water, 0.05:1, 39 °C, 800 W, 47 min	TPC: 119.61 of total peak area (V·s) by HPLC	(Souza et al., 2022)
	Lippia citriodora	MAE	MeOH:water (80:20 v/v), ChCl with LA, tartaric acid, 1,3-butanediol, ethylene glycol, xylitol, 1,2-propanediol, fructose, sucrose, maltose, glucose, urea, (1:1–3:1) with water (0–70 %)	MAE, ChCl:LA (1:2) with 32.19 % of water, 63.68 °C, 17.08 min	TPC: 73.13 mg GAE g^{-1} dw by FC; Iridoids: 9.69 mg g^{-1} by HPLC; Phenylpropanoids: 14.87 mg g^{-1} by HPLC; Flavonoids: 6.28 mg g^{-1} by HPLC; Verbascoside: 12.52 mg g^{-1} by HPLC	(Ivanović et al., 2018)
	Eucommia ulmoides leaves	UAE, HRE, MAE	Methanol:water (50:50, v/v), water, ChCl with L- (+)- ascorbic acid, LA, fructose, glucose, Gly; proline:Gly; CA:maltose, (1:1–4:1) with water (10–100 %) or ethanol (25–100 %)	MAE, ChCl:L-(+)-ascorbic acid (2:1) with 75 % of water, 25 mL g^{-1} liquid-solid ratio, 420 W, 17 s	Iridoids: 8.99 mg g^{-1}; flavonoids: 4.17 mg g^{-1}; phenolic acids: 12.55 mg g^{-1}, by HPLC	(Luo et al., 2022)
	Eucommia ulmoides leaves	UAE, HWE, MAE	Ethanol, water, ChCl with sucrose, glucose, 1,4-butanediol, ethylene glycol, Gly, CA, LA, oxalic acid, (1:1–1:5), with ascorbic acid (0–0.4) and water (0–80 %)	MAE, ChCl:1,4-butanediol, ascorbic acid (1:1:0.20) with 20 % of water, 18.53 mL g^{-1} liquid-solid ratio, 53.03 °C, 20.08 min	Aubucin: 4.288 mg g^{-1}; geniposidic acid: 6.897 mg g^{-1}; geniposide: 0.330 mg g^{-1}; chlorogenic acid: 3.659 mg g^{-1}; rutin: 1.143 mg g^{-1}; isoquercetin: 1.087 mg g^{-1}, by HPLC	(Yu et al., 2021)
	Lonicerae japonicae	HRE, UAE, MAE	ChCl with 1,2-propanediol, Gly, ethyl glycol, 1,3 butanediol, 1,4 butanediol, urea, propanedioic acid, glucose, sorbitol; oxalic acid:sucrose, LA:sucrose and LA:glucose, (1:1–1:7) with water (0–90 %)	MAE, ChCl:1,3-butanediol (1:6) with 10 % of water, 9 mL g^{-1} liquid-solid ratio, 60 °C, 20 min	Chlorogenic acid: 26.07 mg g^{-1}; caffeic acid: 0.148 mg g^{-1}; 3,4-dicaffeoylquinic acid: 0.930 mg g^{-1}; 3,5-dicaffeoylquinic acid: 23.67 mg g^{-1}; 4,5-dicaffeoylquinic acid: 8.85 mg g^{-1}, by HPLC	(Peng et al., 2016)
	Mitragyna speciosa leaves	MAE	CA:glucose (4:1–6:1)	MAE, CA:glucose (5:1), 1:15 w/v, 270 W, 15 min	TPC: 358.59 mg GAE g^{-1} by FC	(Herman et al., 2021)

CA, Citric acid; CE, Catechin equivalents; ChCl, Choline chloride; CUPRAC, Cupric reducing antioxidant capacity; dw, Dry weight; fw, Fresh weight; FC, Folin-Ciocalteu method; GAE, Gallic acid equivalents; Gly, Glycerol; HAE, Homogenization-assisted extraction; HHPAE, High hydrostatic pressure-assisted extraction; HRE, Heat-refluxing extraction; HPLC, High-performance liquid chromatography; HSE, Heating–stirring extraction; HWE, Hot water extraction; LA, Lactic acid; MAE, Microwave-assisted extraction; NADES, Natural deep eutectic solvents; SLE, Solid-liquid extraction; TE, Trolox equivalents; TFC, Total flavonoid content; TPC, Total phenolic content; UAE, Ultrasound-assisted extraction; UPLC, Ultra-performance liquid chromatography.

20 % of water, 80 °C, 800 W and 10 min. Oleuropein was the main phenolic compound identified in both olive leaves and ripe olive extracts. Boli et al. (2022) compared MAE with solid-liquid extraction (SLE) for the extraction of polyphenols from olive leaves using choline chloride with urea and acetic acid as the components of the NADES. MAE with the solvent mixture of choline chloride:acetic acid (1:2) with ethanol:water (80:20 w/w) proved to be highly effective in recovering phenolic compounds, providing extracts with significant antioxidant activity in a very short processing time. In another study, Alañón et al. (2020) optimized the MAE extraction of polyphenols from olive leaves using different combinations of NADES (choline chloride with lactic acid, oxalic acid, tartaric acid, 1,4-butanediol, ethylene glycol, xylitol,

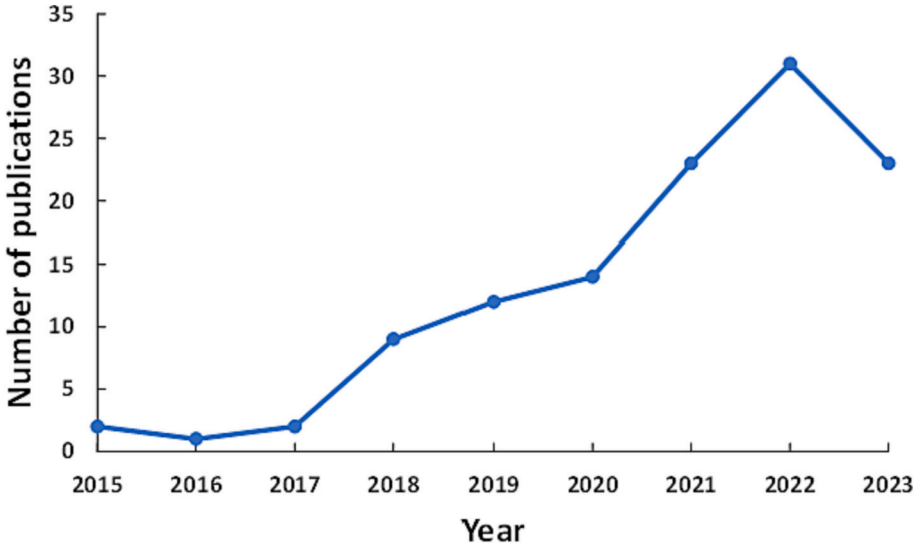

Fig. 3. Number of documents published (until September 2023) concerning microwave-assisted extraction and natural deep eutectic solvents.

Table 2
Scaling of MAE and NADES applications for the recovery of phenolic compounds.

Sample	Scale-up	Extraction Technique	NADES composition	Selected conditions	Polyphenol concentration	Reference
Grape pomace	15 g sample, 500 mL NADES	MAE and UAE (simultaneous)	ChCl:citric acid, 30 % of water	UAE: 500 W, 5 min; MAE: 300 W, 10 min	Anthocyanins: 1.77 mg g dw^{-1} by HPLC	(Panić et al., 2019)
Mitragyna speciosa leaves	50 g sample	Maceration, MAE	ChCl:sorbitol (3 g/g) with water 1:1 v/v	60 % W microwave power, 20 min, 20 mL g^{-1} of liquid-solid ratio	TPC: 526.12 μg GAE g^{-1} by FC	(Prabowo et al., 2022)
Eleutherine bulbosa bulbs	50 g sample	MAE	ChCl:sorbitol (1:1 g/g) with 50 % of water	20 % W, 5 min, 12:1 mL g^{-1} of liquid-solid ratio	TPC: 39.88 mg GAE g^{-1} by FC	(Rijai et al., 2023)
Eleutherine bulbosa bulbs	50 g sample	MAE	Citric acid:glucose (1:1 g/g) with water	270 W, 15 min, 1:8 g/mL solid-liquid ratio	TPC: 61.63 mg GAE g^{-1} by FC	(Yusuf et al., 2021)
Eleutherine bulbosa bulbs	50 g sample	MAE	Lactic acid:sucrose (4:1 g/g) with 30 % of water	360 W, 10 min, 1:10 g/mL of solid-liquid ratio	TPC: 82.43 mg GAE g^{-1} by FC	(Ahmad et al., 2023)
Sea buckthorn leaves	500 g sample	MAE	ChCl:1,4-butanediol (1:3, mol/mol) with 20 % of water	17 min, 64 °C, 21 mL g^{-1} of liquid-solid ratio	Total flavonoids: 20.82 mg g^{-1} by HPLC Individual compounds: Rutin (8.97 mg g^{-1}), quercetin-3-O-glucoside (1.82 mg g^{-1}), quercetin (9.10 mg g^{-1}), kaempferol (0.44 mg g^{-1}), isorhamnetin (0.49 mg g^{-1}), by HPLC	(Cui et al., 2018)
Lettuce sativa, blonde oak leaves	4 kg sample	MAE (solvent-free)	–	4000 W, 30 min, 100 °C	TPC: 6.71 mg/100 g FW by FC Individual compounds: Quercetin derivative (2.4 mg L^{-1}), Flavonoid (4.7 mg L^{-1}), Quercetin-3-O-(6″-malonyl)-glucoside (0.12 mg L^{-1}), Luteolin derivative (5.8 mg L^{-1}), by HPLC	(Périno et al., 2016)

Acronyms are defined in Table 1.

1,2-propanediol, maltose, and urea). Among these combinations, the maximum recovery of phenolic compounds (ca. 29 mg g^{-1}) and oleuropein (11 mg g^{-1}) was achieved using choline chloride:ethylene glycol (1,2) with 43.3 % water content; other conditions were 79.6 °C for 16.7 min for temperature and processing time, respectively.

Oleuropein and hydroxytyrosol have been identified as the main phenolic compounds present in olive pomace extracts (Chanioti and Tzia, 2018). Similarly, oleuropein has been confirmed as the most abundant phenolic compound in olive leaf extracts (Alañón et al., 2020; Bonacci et al., 2020; Boli et al., 2022). Oleuropein and hydroxytyrosol have remarkable pharmacological effects, including antioxidant, anti-inflammatory, anticancer, antiviral, antimicrobial, and antiatherogenic activities (Alañón et al., 2020). These recovered compounds can be used as a substitute for sulfur dioxide in winemaking (Ruiz-Moreno et al., 2015), in food packaging (Luzi et al., 2018; Cejudo Bastante et al., 2018) contributing to the preservation of food products, or as UV filters in cosmetics (Galanakis et al., 2017). Regarding the NADES composition, despite the variety of combinations tested by the different authors, choline chloride and polyols, with a low percentage of water to improve the working properties, seem to be the most appropriate options. In any case, MAE processing significantly reduced the working time compared to other extractive techniques.

2.2. Winery waste

Wine production is another important agricultural and industrial activity, with *Vitis vinifera* being the most cultivated species for wine production. Around winemaking processes, which involves the processing of >60 million tons of grapes per year (Tapia-Quirós et al., 2022b; Tapia-Quirós et al., 2022c; Barba et al., 2016; Teixeira et al.,

2014), almost 20 million tons of winery by-products are discarded yearly, such as grape pomace (skin and seeds), steams, wine lees, and wastewater (Melo et al., 2015). One ton of grapes generates about 0.13 t of pomace, 0.03 t of stems, 0.06 t of lees, and 1.65 m^3 of wastewater (Barba et al., 2016; Pérez-Bibbins et al., 2015). Composting is the most common destination when recycling this type of waste. Anyway, as pointed out elsewhere (Tapia-Quirós et al., 2022b; Berbel and Posadillo, 2018), they result in an exceptional source of building blocks for the chemical industry; besides, winemaking waste is rich in polyphenols. A comprehensive characterization of the phenolic profile of winemaking waste can be found elsewhere (Mir-Cerdà et al., 2023b).

For example, phenolic compounds like gallic acid, syringic acid, and hesperidin can be recovered from these residues (Tapia-Quirós et al., 2022a). Gallic acid is a natural endogenous hydroxybenzoic acid effective in treating metabolic disorders, including obesity, liver steatosis, and diabetes (Behera et al., 2023). Gallic acid can act as an anti-obesity agent by inhibiting oxidative and inflammatory pathways in obesity (Behera et al., 2023). Syringic acid is also a hydroxybenzoic acid with interesting properties in the biomedical and industrial sectors. Among them, caries-reducing properties and thus it has been employed in the preparation of dental cement (Srinivasulu et al., 2018). Hesperidin is a flavonoid with a well-known potent antiviral agent. Recently, it has been demonstrated that hesperidin easily binds to key proteins of the SARS-CoV-2 thus blocking its replication (Montenegro-Landívar et al., 2021a). The wine matrices are also highly appreciated and recognized for their content in rutinosides used in nutraceuticals to improve venous insufficiencies or stilbenes with attributed rejuvenating activity (Vidal-Casanella et al., 2021).

Papers published to valorize wine waste through MAE and NADES are still scarce. Cvjetko Bubalo et al. (2016) evaluated MAE for the extraction of polyphenols from grape skins using this type of solvent. Choline chloride:oxalic acid with 25 % water was selected as the most effective solvent in the extraction of grape skin phenolic compounds compared to conventional organic solvents. MAE results in better extraction performances than SLE or UAE counterparts. After evaluating MAE conditions, it was concluded that an optimal temperature of 65 °C yielded the best results. Additionally, the extraction efficiency improved as the irradiation time increased, up to 50 min. However, a further increase in extraction time resulted in a decline in extraction yield. This decrease can be attributed to thermal degradation and oxidation of the target compounds, indicating the need to avoid excessive exposure to microwave energy.

2.3. Coffee waste

Coffee stands as one of the most consumed beverages worldwide, with global consumption of 2.25 billion cups per day (Tzani et al., 2023). In this way, the production of coffee has increased since 2010, going from 140 to 152 million/year (López-Linares et al., 2021a). Therefore, a wide range of waste is obtained during its production (e.g., husk, pulp, mucilage, coffee silver skin, and spent coffee grounds). The most significant residue generated is spent coffee grounds (6 million metric tons per year), then coffee pulp and husk with 0.50 and 0.18 t t^{-1} of fresh coffee, respectively (López-Linares et al., 2021a). These residues, in turn, are a valuable source of bioactive compounds, such as phenolic acids (e.g., chlorogenic, caffeic, ellagic, trans-ferulic, feruloyl quinic, gallic, *p*-hydroxybenzoic, *p*-coumaric, *p*-coumaroyl quinic, protocatechuic and tannic acids), flavonoids (e.g., catechin, epicatechin, rutin, and quercetin), trigonelline, and caffeine, among others (López-Linares et al., 2021a). However, currently, most of this waste is recycled for composting, so most bioactives are not used to obtain high value-added products. For instance, caffeine, in addition to its stimulating action on the central nervous system, stands out for activating fat metabolism, so it is included in numerous anti-cellulite cosmetic preparations. Quantitatively, derivatives of caffeic acid and catechins that combat the effects of free radicals in the body are also important.

Thus, López-Linares et al. (2021a) identified spent coffee grounds as a potential resource within the biorefinery context. They optimized the extraction of phenolic compounds using MAE with NADES and obtained a maximum extraction yield (0.48 mg GAE g^{-1}) with choline chloride: glycerol (0.53 M ratio, 20 % water), at 120 °C for 15 min. In addition, they produced biobutanol from the residual NADES extracted from the spent coffee grounds. Also, Tzani et al. (2023) optimized the extraction of phenolic compounds from spent coffee grounds using MAE with NADES. They found that betaine:glycerol (1:3) with water (50 % v/v) was an excellent extraction media when applied in MAE under the optimized conditions (90 °C, 60 min, and 1 % solid-to-liquid ratio). This resulted in the extraction of total phenolic content (TPC) and total flavonoid content (TFC) values up to 30.9 mg GAE g^{-1} and 22.8 mg of catechin equivalent per gram (mg CATE g^{-1}), respectively.

2.4. Beer waste

Beer is the fifth most frequently consumed drink worldwide, with a production in 2018 of about 191.1 million kL (Olivares-Galván et al., 2022). Since beer is a fermented beverage produced from malted cereal, water, yeast, and hops, the main waste generated are brewer's spent grain, barley malt rootlets, spent hops, and spent yeast (Olivares-Galván et al., 2022). Brewer's spent grain is the most abundant residue from brewing, and an average of 20 kg of this residue is generated per 100 L of beer; barley malt rootlets usually constitute around 3–5 % of the initial total barley weight; spent hops constitute 0.2–0.4 % of the wort; and brewer's spent yeast is the second most abundant residue from brewing (10–15 %) (Olivares-Galván et al., 2022; López-Linares et al., 2021b).

Brewer's spent grain represents an interesting lignocellulosic residue due to its high content of phenolic compounds, especially hydroxycinnamic acids (such as ferulic, *p*-coumaric, sinapic, and caffeic acids), and hydroxybenzoic acids in a lesser extend (e.g., syringic acid) (López-Linares et al., 2021b). For the above, López-Linares et al. (2021b) studied the extraction of phenolic compounds from brewer's spent grain, using NADES and MAE. As findings, choline chloride:glycerol with 37.46 % of water (v/v) was the most effective NADES, far exceeding the capacity of methanol:water as a conventional solvent (80 % v/v); a phenolic compounds recovery of 2.89 mg GAE g^{-1} was obtained under the optimized conditions of 100 °C and 13.30 min of extraction time.

2.5. Fruit and vegetable waste

The vegetable and fruit sector generates around 90 million tons of residues per year in Europe which represent an important problem of waste disposal (Montenegro-Landívar et al., 2021b). The waste includes trimmings, peelings, stems, seeds, shells, bran, and other residues after the juice extraction, oil, starch, and sugar (Kumar et al., 2017). These wastes are rich in several bioactive components, including phenolic compounds like resveratrol, luteolin, quercetin, hesperidin, and naringenin, all displaying remarkable antioxidant activity (Montenegro-Landívar et al., 2021a).

As examples dealing with phenolic compounds recovered from fruit residues, Gao et al. (2020) studied mulberry leaves comparing different NADES and extraction techniques, such as heat-refluxing extraction (HRE), UAE, and MAE. As a result, chloride:glycerol (1:2) with 20 % water resulted in the best NADES option. Furthermore, MAE was the most efficient extraction technique under the optimized conditions of 66 °C, 18 min, and 1:20 w/v solid-liquid ratio, with a polyphenol recovery, determined by HPLC, of ca. 8.4 mg g^{-1}. González-Rivera et al. (2021), combined acid-based NADES and MAE to improve the extraction of phenolic compounds from chestnut shells. Choline chloride:oxalic acid featured high biomass dissolution ability, good response to microwave irradiation and best yields of phenolic compounds. The optimized MAE method recovered 295.2 mg GAE g^{-1} dw of total phenolic compounds under the selected experimental conditions of 1:10 w/v, 60 min, and 85 °C. In another study, Sharma and Dash (2021) optimized the

extraction of phenolic compounds from black jamun (*Syzygium cumini*) pulp using MAE with a NADES based on choline chloride:citric acid, with different percentages of water. As findings, the water content in the NADES (40 % of water) and microwave power (400 W) had a positive impact on total phenolic (45 mg GAE g^{-1}) and anthocyanin (9.3 mg C3G g^{-1}) recovery, using a solvent/sample ratio of 48 mL g^{-1} and 240 s of extraction time. In another case, a ternary NADES combination of glycerol:xylitol:fructose (1:1:1 M ration) with 20 % water enhanced the extraction yield of five target compounds (caffeoyl malic acid, psoralic acid-glucoside, rutin, psoralen and bergapten) from fig leaves, being MAE more effective than UAE under the selected extraction conditions (64.46 °C, 24.43 min, liquid-solid ratio 17.53 mL g^{-1}) (Wang et al., 2017). This study also demonstrated the better performance of NADES compared to methanol:water mixtures.

Regarding vegetable residues, Wei et al. (2015) investigated fourteen NADES systems for the extraction of phenolic compounds from *Cajanus cajan* leaves by MAE. A NADES consisting of choline chloride:maltose (1:2) with 20 % water presented the best extractability for both polar and weak polar compounds compared to conventional solvents. Moreover, the influential parameters of MAE were optimized, and the following conditions were chosen: 60 °C of processing temperature, 12 min of extraction time, and 30:1 mL g^{-1} of solvent/sample ratio. Likewise, Pal and Jadeja (2019a) optimized MAE for the recovery of phenolic compounds from onion peel waste employing choline chloride: urea:water (1:2:4). MAE extracted the highest amount of phenolic compounds (80.45 mg GAE g^{-1} dw) with 12-fold reduction in extraction time (compared with heating–stirring and Soxhlet extraction), under the optimized conditions (100 W, 15.03 min, 54.97 mL g^{-1}).

2.6. Plant waste

Medicinal plants have been used for centuries as remedies in traditional medicine in several forms, including unprocessed products, mixtures of different species, dietary supplements, nutraceuticals, and phytopharmaceuticals (Montenegro-Landívar et al., 2021a; Chiriac et al., 2021). Noticeable amounts of waste rich in active components are generated around the plant collection and processing, so can be used to recover target compounds. Since the development of new formulations with integrated bioactive compounds from natural sources is currently of great interest to the pharmaceutical industry, the assessment of the extraction and identification of chemical components of plants is essential (Chiriac et al., 2021).

Various relevant studies combining MAE with NADES have been reported in the scientific literature. For instance, Souza et al. (2022) recovered phenolics from leaves of *Eugenia uniflora* L. Choline chloride: lactic acid 1:3 (mol/mol) with 20 % water (w/w) was selected from five different NADES compositions. Under the optimized MAE conditions (0.05:1 (w/w) plant/NADES ratio, 47 min of extraction time, 39 °C, and 800 W), NADES showed a superior performance than the conventional alternative using ethanol:water 7:3 (v/v). In another study, Ivanović et al. (2018) tested 11 different NADES and optimized MAE factors to recover bioactive compounds from *Lippia citriodora*. Choline chloride: lactic acid exhibited the highest extraction yield for TPC (73.13 mg g^{-1}), iridoids (9.69 mg g^{-1}), phenylpropanoids (14.87 mg g^{-1}), flavonoids (6.28 mg g^{-1}), and verbascoside (12.52 mg g^{-1}), being higher values than those with methanol under optimal MAE conditions (63.68 °C, 32.19 % of water, and 17.08 min of extraction time). They also found that temperature and water content showed a higher effect on the extraction of polyphenols than irradiation time.

2.7. Scaling-up cases

As mentioned, most of the applications highlighted in this review deal with the topic at a laboratory scale, where the advantages of the combination of MAE and NADES are quite evident. Table 2 details the scaling works published to date. Panic et al. deal with the grape pomace treatment in a 0.5 L batch reactor combining MAE and UAE as extractive technologies and using choline:citric acid NADES (Panić et al., 2019). The study focuses on the recovery of anthocyanins as the reddish pigments of this type of matrices. Other studies focus on treating leaves or bulbs of medicinal plants (e.g., *Mitragyna speciosa* and *Eleutherine bulbosa*) to obtain extracts rich in antioxidants; the global antioxidant capacity using FC assesses the performance of the procedures (Prabowo et al., 2022; Rijai et al., 2023; Yusuf et al., 2021; Ahmad et al., 2023; Cui et al., 2018) to process ca. 50 g of sample per batch. Progressing in the scaling, a system was developed to extract flavonoids from *Hippophae rhamnoides* (sea buckthorn). The device allowed treating 0.5 kg of the sample, obtaining quantities >20 mg kg of flavonoids, the most abundant being quercetin and some of its glycosides such as rutin. In our search, the larger system corresponded to a microwave reactor for up to 4 kg of sample, used to recover polyphenols from lettuce (Périno et al., 2016). The phenolic concentration was estimated by FC and the most abundant flavonoids were determined by HPLC. Although no solvent was used in this case, it could be a good starting point for implementing MAE and NADES on a larger scale.

From the point of view of practical applicability, the use of NADES in scaling does not entail any additional challenge other than the desirable recovery and reuse of the solvent for future extractions (see Section 3). From the point of view of microwave technology, the principal challenges concern the development of equipment for processing large quantities of samples, especially for its implementation on a pilot and industrial plant scale.

2.8. Overall remarks

As a general comment of this section, the main advantages of MAE deal with speed and efficiency, leading to higher extraction yields in a shorter time. However, it can cause a parallel degradation of sensitive compounds. On the other hand, NADES are aligned with green chemistry principles because they come from natural sources, are biodegradable, and can be designed with tailored properties, usually providing efficient extraction yields. There is still limited knowledge about NADES behavior so more fundamental research is needed to dig for affinity and specificity features. A broad variety of NADES compositions have been proposed depending on the cases, in line with the disparity of matrices mentioned. Combining MAE and NADES is a green synergic alternative that joints extraction efficiency of MAE and NADES with MAE speed. From this approach, the selective extraction of individual polyphenols remains a very challenging objective, requiring further purification steps, such as membrane-based or adsorption/desorption processes discussed in Section 3.

Going deeper into the issue of the composition of NADES and its capacity and selectivity for the recovery of phenolic compounds, from the analysis of the information in Table S1, we conclude that choline chloride is the quasi-universal acceptor (few studies mention other acceptors such as betaine (Tzani et al., 2023) or ethanolamine (Shang et al., 2022)). As a general guideline, glycerol and, more occasionally, other diols and polyols (e.g., ethylene glycol and butanediol) seem to be the most popular options to achieve a general recovery of polyphenolic compounds, without going into the particularities of individual compounds, thus obtaining extracts with high overall antioxidant capacity but without selectivity towards the different families. Perhaps the matrix nature is also influential in the choice of the donor agent, but more studies are needed to obtain knowledge on this topic. When dealing with the different phenolic families, phenylethanoids and simple phenolic acids are mostly extracted with glycerol as a donor in the NADES formulation. However, for quinic acid conjugates and condensed species, a donor acid (e.g., lactic or malic acid) is usually incorporated (Ivanović et al., 2018; Popovic et al., 2021). Free flavonoids (aglycones) are typically recovered including polyols in the NADES composition while glycosides from different subfamilies, such as flavonols, flavones, or anthocyanins glycosides, are generally better extracted using donor

acids (e.g., citric, lactic, or oxalic) (Chanioti and Tzia, 2018; Cvjetko Bubalo et al., 2016; Souza et al., 2022; Popovic et al., 2021); ascorbic acid is even used in several cases, which can also have a protective effect by minimizing the oxidation of the analytes (Luo et al., 2022; Yu et al., 2021).

The comparison of the performance of MAE with alternative extractive techniques in the context of the study topic, i.e., focused on NADES and recovery of phenolic compounds in agri-food matrices, has been carried out in various studies presented in Table 1. As shown, techniques such as SLE, UAE, HAE, HHPAE, and HRE have been contrasted with MAE to investigate the best options. In general, MAE has proven to be a powerful option, resulting in the technique of choice in most cases. However, depending on the situation, some authors have preferred alternatives based on additional criteria other than the extractive performance, such as simplicity, ease of scaling, energy consumption, etc. Traditional SLE (e.g., by maceration or assisted by mechanical stirring or heating) is a reliable option, although it may require longer extraction times than MAE. Although MAE is often preferred at the laboratory scale (Boli et al., 2022; Pal and Jadeja, 2019a,b; Popovic et al., 2021; Yao et al., 2015), SLE-based techniques stand out at the industrial level. Besides, extraction conditions are energetically milder, thus preventing the degradation of labile compounds. With a more advanced SLE technology such as HAE, the greater shaking efficiency provides high extractive yields with relatively short process times of about 30 min (Chanioti et al., 2021; Chanioti and Tzia, 2018). However, the use of HAE is not always chosen and, for example, MAE has been recommended for the extraction of phenolic compounds from mango skin (Pal and Jadeja, 2019b), sour cherry pomace (Popovic et al., 2021), onion skin (Pal and Jadeja, 2019a), or various medicinal plants (Yao et al., 2015; Peng et al., 2016). UAE is a very efficient approach when working with conventional solvents. In the case of NADES, however, this technology is less suitable since the high viscosity of the medium hinders the cavitation and, thus, the analyte extraction. As shown in Table 1, MAE is often preferred over UAE (Yao et al., 2015; Tzani et al., 2023; Gao et al., 2020; Wang et al., 2017; Popovic et al., 2021; Luo et al., 2022; Yu et al., 2021; Peng et al., 2016) for lab scale applications. UAE has been chosen only in some specific cases, such as for extracting anthocyanins from grape skins (Cvjetko Bubalo et al., 2016). Other technologies, such as HHPAE and HRE, have also been compared with MAE, although they have not provided better results (Gao et al., 2020; Luo et al., 2022; Peng et al., 2016). To date, no studies have been found comparing MAE-NADES with enzyme-assisted, supercritical fluid, subcritical fluid, and pulse electric field-assisted extraction to recover phenolic compounds. Finally, regarding the extractive capacity of NADES, from Table 1, we deduce that the quantitative performance always favors NADES when compared with conventional solvents. Hence, hydro-organic solvents or water have been chosen occasionally because of cost, sustainability, greenness, or other criteria.

3. Purification of the extracts and NADES removal/recovery

Once the successful extraction of polyphenols with NADES has been proved, the next scientific challenge lies in separating the polyphenols from the NADES solution, to yield products with high added value. Simultaneously, within the framework of sustainability and green chemistry, the recovery and reuse of NADES to process a new batch of waste are crucial.

Alongside the extraction of phenolic compounds present in the range of matrices indicated in the previous section, other (undesired) compounds are often co-extracted; hence, their separation in a further purification stage is necessary. In addition to removing these interfering species, this stage also aims to pre-concentrate the compounds of interest. This can be achieved by membrane separation and adsorption-desorption processes, resulting in a purified polyphenol product of higher quality and added value, suitable for the intended applications in the food, cosmetic, and pharmaceutical industries (Tapia-Quirós et al., 2022b).

Membrane separation processes are the main nondestructive physicochemical techniques to separate macromolecules and small molecules from agri-food industry waste (Cassano et al., 2016; Tapia-Quirós et al., 2022d). Membranes act as selective barriers, allowing the passage of compounds based on their solute properties and the characteristics of the active layer. Hence, molecular weight cut-off (MWCO), solute size, charge, and interactions with the membrane surface are often significant factors in the optimization (Tapia-Quirós et al., 2022b; Zagklis and Paraskeva, 2015). Membrane operations separate a feed solution into two fractions, that are the permeate or filtrate and the retentate or concentrate. The permeate comprises the solvent and solutes that successfully pass through the membrane. The retentate or concentrate, on the other hand, contains particles and dissolved compounds that are partially retained by the membrane (Tapia-Quirós et al., 2022b). The most important pressure-driven membrane techniques are microfiltration (MF 0.1–5 μm, 1–10 bar), ultrafiltration (UF 0.5–100 nm, 1–10 bar), nanofiltration (NF 5–10 nm, 10–30 bar), and reverse osmosis (RO $<$ 0.5 nm, 35–100 bar) (Tapia-Quirós et al., 2022c; Tapia-Quirós et al., 2022d). The values in parentheses correspond to the pore sizes and the pressures necessary for each process. Other membrane processes can be categorized based on concentration gradient-driven processes (e.g., dialysis), and electrical potential-driven processes (e.g., electrodialysis). To date, membrane technologies have been extensively and successfully applied to treat conventional aqueous and hydro-organic extracts. Studies on the filtration of NADES extracts are still very scarce (Liang et al., 2018; Liang and Guo, 2022; Gholami et al., 2022; Ippolitov et al., 2022; Kim et al., 2018; Wahlström et al., 2017; Zhang et al., 2022; Mahto et al., 2017). For instance, ultrafiltration and electrodialysis were used to recover cellulose, hemicellulose, lignin, as well as NADES, from *Eucalyptus globulus* wood extracts, using choline chloride:ethylene glycol (2:1) (Liang et al., 2018). Also, ultrafiltration and bipolar membrane electrodialysis were used to recover cellulose, hemicellulose, lignin, and NADES from rice straw extracts, using choline chloride:ethylene glycol (1:2) (Liang and Guo, 2022), and lactic acid:ethylene glycol (1:1) (Zhang et al., 2022). On the other hand, in the treatment of birch wood (Ippolitov et al., 2022) and switchgrass (Kim et al., 2018) biomass, ultrafiltration was employed to recover lignin and the constituents of the NADES (choline chloride:lactic acid (1:10) and choline chloride:*p*-coumaric acid (1:1)). However, to the best of our knowledge, we have not found any application to polyphenolic NADES extracts from agri-food waste, probably because of the novelty and complexity of this issue.

Adsorption-desorption processes allow for simultaneously recovering, purifying, and concentrating target compounds, thereby obtaining a final product that is rich in phenolic compounds. During the adsorption process, the sorbent (e.g., the resin) interacts with molecules in the liquid phase through Van der Waals forces, hydrogen bonds, ionic interactions, or hydrophobic interactions. Reversible adsorption is essential to facilitate the recovery of the solute once is retained in the sorbent (Tapia-Quirós et al., 2022b; Pérez-Larrán et al., 2018). In general, resins are synthetic polymeric materials encompassing various materials like polystyrene-divinylbenzene copolymers, polymethacrylate, and more (Tapia-Quirós et al., 2022b; Soto et al., 2011). They can be modified with ion-exchange groups for specific applications. The adsorption characteristics of resins depend on factors such as polymer composition, pore structure, and surface properties (Tapia-Quirós et al., 2022b; Wang et al., 2019). As can be seen in an interesting bibliographical review, several studies have been published on the application of resins to recover polyphenols using conventional solvents (dos Santos et al., 2022). Nevertheless, there is a very scarce number of publications on the use of NADES and resins (Gao et al., 2020; Wang et al., 2017; Panić et al., 2019; Panić et al., 2021; Rodríguez-Juan et al., 2021; Grillo et al., 2020). For instance, the recovery of target phenolic compounds was performed by resins like Sepabeads SP825L for blueberry-peel extracts (choline chloride:lactic acid (1:1) with 22 % of water) (Grillo et al., 2020); Sepabeads SP825L for orange peel extracts

(choline chloride:ethylene glycol with 50 % of water) (Panić et al., 2021); Sepabeads SP207 for grape pomace extracts (choline chloride: citric acid (2:1) with 30 % of water) (Panić et al., 2019); D101 macroporous resin for fig leaf extracts (glycerol:xylitol:D-(−)-fructose (3:3:3) with 20 % of water) (Wang et al., 2017), Amberlite XAD-16 for virgin olive oil extracts (choline chloride:xylitol:water (2:1:3)) (Rodríguez-Juan et al., 2021), and NKA-9 for mulberry leaves extracts (choline chloride:glycerol (1:2) with 20 % of water) (Gao et al., 2020). In general, these papers use non-functionalized resins to retain efficiently a large percentage of the solutes of interest (while the NADES are mainly unretained) in a batch format. Subsequently, the resins are washed with aqueous solutions to eliminate the residual amounts of retained NADES. Finally, the phenolic compounds are eluted with a solvent with a high percentage of alcohol. From a pilot plant and industrial point of view, the resin technology is implemented in column.

3.1. NADES recycling

As discussed throughout this review, NADES have been proposed as potential green alternatives to organic solvents in extraction processes; however, in line with the intended green nature of the waste processing methodologies, the recycling and reuse of NADES seem to be a crucial issue that presents significant challenges for sustainable, environmentally friendly, and cost-efficient industrial applications, as reported in the recent review by Isci and Kaltschmitt (2022). Different technologies have been investigated for the recovery and purification of NADES, such as anti-solvent addition, recrystallization, liquid–liquid extraction, solid–liquid extraction, short path distillation, supercritical fluid extraction, density-based separations, and membrane filtration (Isci and Kaltschmitt, 2022). Most of the strategies discussed below refer to the recovery of NADES from extracts obtained using different extraction techniques (not necessarily MAE) but they can be perfectly extrapolated to MAE without further considerations.

Anti-solvent processing consists of adding an anti-solvent (e.g., water, ethanol, or acetone) at high concentrations, which causes the breaking of hydrogen bonds between the NADES components and precipitates the solubilized solids from the mixture (Isci and Kaltschmitt, 2022; Kumar et al., 2016; Wang et al., 2020; Huang et al., 2017; Chen et al., 2018). This enables the separation of the target compounds from the NADES. Afterwards, the anti-solvent is removed by evaporation, and the remaining NADES is recycled within the process (Isci and Kaltschmitt, 2022). For instance, water was used as an anti-solvent for NADES recovery in studies for lignin extraction from rice straw (Kumar et al., 2016), cellulose extraction from cotton fibers (Wang et al., 2020), and rutin extraction from tartary buckwheat hull (Huang et al., 2017). Ethanol was another efficient anti-solvent for NADES recovery in studies for lignin recovery from switchgrass (Chen et al., 2018). Acetone was used to precipitate NADES constituents from the extraction of biodiesel from soybean oil (Homan et al., 2017). In another application, water and acetone were used to recover NADES from beech wood sawdust treatment for the lignocellulosic biomass fractionation (Mamilla et al., 2018).

Liquid-liquid extraction separates the NADES solutes between two immiscible solvents (the NADES and the organic solvent). Afterward, the two liquid phases can be easily separated by decantation (Isci and Kaltschmitt, 2022; Zhang and Yu, 2013; Xu et al., 2019; Smink et al., 2020). For instance, a two-step liquid-liquid extraction was used to recover flavonoids and NADES (choline chloride:levulinic acid:*N*-methyl urea) from citrus peel waste using ethyl acetate (1:1 v/v) and n-butanol (1:1 v/v) (Xu et al., 2019). Also, metal chloride was supplemented for recycling NADES (choline chloride:citric acid:water) used to convert xylan and xylose from hemicellulose fraction into furfural (Zhang and Yu, 2013).

Solid-liquid extraction using macroporous resins has also been proposed for NADES recovery. The NADES extracts are fed into a column with a macro-porous resin. The target compounds are adsorbed by the resins while the NADES is washed out using water. Afterwards, a desorbing solvent (e.g., ethanol) is required to elute the retained compounds. The recycled NADES should be evaporated for water removal and then could be reused in the system (Isci and Kaltschmitt, 2022; Gao et al., 2020; Wang et al., 2017; Panić et al., 2019; Grillo et al., 2020; Liu et al., 2019). Some authors utilized this technique to enrich and separate target compounds (e.g., polyphenols) from NADES extracts of mulberry (*Morus alba*) leaves (Gao et al., 2020), fig (*Ficus carica*) leaves (Wang et al., 2017), grape pomace (Panić et al., 2019), turmeric (*Curcuma longa*) (Liu et al., 2019), and blueberry peel (Grillo et al., 2020). The recovered NADES exhibited a good performance when applied to new extraction cycles.

Short-path distillation is a rapid separation technique that involves exposing the distillate to high temperatures for a brief period (a few seconds to tens of seconds) and covering a short distance (Kleiner et al., 2016) from the evaporator to the condenser. This process is commonly conducted under reduced pressures to separate heat-sensitive compounds. The application of this method for recovering NADES has not been extensively investigated yet (Isci and Kaltschmitt, 2022; Cvengroš et al., 2000). Kleiner et al. (2016) used short path distillation to separate the NADES (choline chloride:glycerol) used in biodiesel production in a single distillation step; in this way, NADES was reused five times without undergoing a noticeable loss of efficacy.

Supercritical fluid extraction uses supercritical fluids (e.g., carbon dioxide) that exceed their critical values in terms of pressure and temperature. They exhibit gas-like viscosity and diffusivity, along with liquid-like density and solvating properties, making them suitable for diverse applications. Supercritical fluid extraction offers a significant advantage since the product can be easily separated and dried through simple expansion. Additionally, the gas used in the process can be recovered, recycled, and reused without requiring additional purification steps (Isci and Kaltschmitt, 2022; Knez et al., 2014; Zhou et al., 2018; Plaza et al., 2020). Plaza et al. (2020) used supercritical CO_2 (100 bar, 35 °C, 1 h) as a stripping phase to recover hydroxytyrosol, tyrosol, and oleuropein from olive mill waste and olive leaves from the NADES extracts (choline chloride:ethylene glycol). The supercritical fluid process not only enabled the recovery of the target compounds but also facilitated the regeneration of the NADES, thereby enabling its recycling within the system.

Membrane-based processes offer great flexibility for selective separation of solutes and recycling of the permeate. Ultrafiltration, electrodialysis, and bipolar membrane electrodialysis techniques are used to recover the NADES constituents from extracts. For example, Ippolitov et al. (2022) employed ultrafiltration to purify spent NADES (choline chloride:lactic acid, 1:10) used in the extraction of lignin from lignocellulosic biomass. They found that the regenerated cellulose membranes RC70PP and Ultracel 5 kDa UF membranes, could be used in the treatment of the spent NADES, and that the addition of ethanol as cosolvent to the spent NADES decreased solvent's viscosity, which improved filtration. Also, Liang et al. (2018) combined ultrafiltration and electrodialysis to separate and recover the NADES (choline chloride: ethylene glycol, 2:1) after biomass fractionation from *Eucalyptus globulus* wood extracts. As findings, electrodialysis after ultrafiltration was proven effective to separate and recover the choline chloride (permeate) and ethylene glycol (retentate). The recovery ratio was 92 % and 96 % for choline chloride and ethylene glycol, respectively, and purities reached 98–99 % after electrodialysis treatment. Furthermore, Liang and Guo (2022) studied the recovery and regeneration of NADES (choline chloride:ethylene glycol, 1:2) using bipolar membrane electrodialysis with ultrafiltration, after biomass pretreatment of rice straw extracts. They obtained a recovery ratio of NADES reached 97.4 %, and the minimum specific energy consumption of NADES recovery approached 6.0 kW h/kg.

The options described in this section are promising, but further studies are needed to clarify the pros and cons of each possibility and understand the most critical factors for NADES recycling and reuse.

4. Conclusions

The combination of MAE with NADES is a novel and advantageous approach for the recovery of polyphenols from different agrifood matrices. The microwave energy enhances the extraction process by providing rapid and efficient heating, and promoting cell wall rupture, resulting in shorter processing time and higher yield compared to other processing technologies. Additionally, NADES offers a greener alternative to traditional organic solvents. NADES come from natural sources and have low toxicity, making them more environmentally friendly and potentially safer for operators. In line with these features, the final products are fully compatible with food, cosmetic, and pharmaceutical applications.

NADES and MAE conditions can be optimized by selecting the appropriate solvent composition (e.g., donor, acceptor, and water percentage) and extraction conditions (such as temperature, time, and power) to recover phenolic compounds. The extraction parameters can be fine-tuned to maximize the extraction efficiency while minimizing potential degradation or alteration of the target compounds. Overall, MAE with NADES represents a promising and environmentally friendly strategy to extract polyphenols offering remarkable advantages in terms of extraction efficiency, time, and environmental sustainability. However, it is important to note that this technique is relatively new, and further research is still needed to fully understand and optimize the process parameters and evaluate the quality and stability of the extracted polyphenols. In our opinion, the MAE-NADES marriage is fairly consolidated at the laboratory level, providing generally superior performance to other technologies. However, the applications developed at the pilot plant level are still very scarce. As reported, the sample quantities processed in scaled-up facilities range from dozens of grams to several kilos, so there is still a long way to go before the technology is available on an industrial level.

Focusing on qualitative aspects, this review demonstrates that the recovery of phenolic compounds by NADES and MAE is generally better than other available combinations. In our opinion, beyond the undoubted extractive efficiency, the weakest points are the purification of the extracts, together with the NADES/target compound separation and the NADES recycling. These last two issues are essential in the line of green chemistry principles. In this regard, anti-solvent techniques provide more complex results while membrane-based techniques have to overcome some technical drawbacks related to the processing of viscous solutions that may potentially damage the membrane integrity. Currently, the most viable purification option relies on adsorption resins that allow the separation and purification of the compounds. At the same time, the NADES can be directly recovered and reused.

The MAE-NADES approach, which combines green solvents and an efficient extraction technique, is aligned with the sustainability principles and multiple Sustainable Development Goals. It directly or indirectly addresses, within the scope of the circular economy, issues related to health (SDG 3), environmental conservation (SDG 13 and 15), and food waste recycling and innovation (SDG 12).

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CRediT authorship contribution statement

Paulina Tapia-Quirós: Data curation, Methodology, Writing – original draft, Writing – review & editing. **Mercè Granados:** Conceptualization, Funding acquisition, Investigation, Methodology, Supervision, Writing – original draft, Writing – review & editing. **Sonia Sentellas:** Conceptualization, Methodology, Supervision, Writing – original draft, Writing – review & editing. **Javier Saurina:** Conceptualization, Funding acquisition, Methodology, Supervision, Writing – original draft, Writing – review & editing.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Javier Saurina reports financial support was provided by Spain Ministry of Science and Innovation.

Data availability

No data was used for the research described in the article.

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