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Sustainable raw materials for efficient valorization and recovery of bioactive compounds

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Abstract

Traditionally used natural resources such as medicinal and aromatic plants are not fully valorized by modern industry which is followed by huge amount of agricultural waste, by-products and co-products. These streams could be a remarkable source of a wide range of bioactive compounds (BACs) (e.g. polyphenols, carotenoids, etc.), however, novel technological procedures for their isolation should be established. Threfore, valorization of under-utilized medicinal plants (medicinal plants from Serbia), food industry by-products (cakes obtained from oilseeds and herbal dust) and agricultural waste as raw material for recovery of BACs with high potential for utilization in pharmaceutical, cosmetic and food sector is in the great focus of investigation in last decade. The aim of this review was to highlight the recent research focused on the valorization of selected side streams and the posibilites of their utilization in isolation of various BACs. Special emphasis was put on challenges, limitations and opportunities associated with the extraction of BACs and further addition of novel extracts in the final products. Present data could be used as a guidelines for further utilization of novel raw materials in production of BACs based on their avalibility, sustainability, chemical profile and bioactive potential.

Key words: By-produts; Agricultural waste; Novel extraction techniques; Bioactive compounds; Valorization

1. Introduction

Food industry and agriculture are recognized as one of the most prominent industry branches in Europe. The main issue in the food production chain is the amount of loss and waste which is estimated at 54% of total loss and waste as upstream processes (including production and postharvest) and 46% of total loss and waste as downstream processes (including processing, distribution, and consumption) (Gustavsson et al., 2011). Therefore, the latest strategy is requested to develop national food waste prevention plans, aimed to reach the objective to reduce food waste by at least 30% by the end of 2025 (Otles et al., 2015).

Traditionally used natural resources such as medicinal and aromatic plants are not fully valorized by modern food, cosmetic and pharmaceutical industry. Based on this, large amount of of agricultural waste, by-products and co-products is being generated. This could be partly attributed to the lack of knowledge on chemical profile and bioactive potential of under-utilized plant species. However, degradation of natural resources worldwide caused by decline in land, water, and biodiversity creates critical concerns about meeting the future demands on global level. Furthermore, modern pharmaceutical and food industries underutilize by-products of their technological processes generating large amount of bio-waste. Disposal of these matters is problematic regarding the safety of property, human health and environmental pollution. Therefore, special measures for the proper waste and by-products disposal are needed, which imposes additional costs and price increase of the main product and later decreases its competitiveness on the market.

It is evident that insufficient use of by-products and waste is the consequence of many factors that is quite difficult to solve, because even if it is found and proved that these streams contain biologically active compounds (BACs) or groups of substances with economic potential, there are no adequate procedures of their isolation. The potential of agri-food wastes and side streams to create new opportunities for high-value products has been underestimated and overlooked. However, valorization of by-products and waste streams through the recovery of its valuable BACs has been recognized as an important way of increasing the sustainability in production in order to create new opportunities on the market. Even though, there has been an extensive work done and published in valorization of various by-products, some important aspects, such as application, safety issues, sensory properties, were lacking and special emphasis should be put in that direction. Therefore, the main goal of this work was to provide in-depth review on the valorization of afore mentioned side streams and the posibilites of their utilization in isolation of various BACs. Present data could be used as a guidelines for further utilization of novel raw materials in production of BACs based on their avalibility, sustainability, chemical profile and bioactive potential.

2. Valorization of sustainable raw materials

The main issues associated with common valorization approach are disadvantages and limitations of traditional extraction procedures which do not satisfy the economic aspects of production in terms of energy and resource preservation, as well as improved yield, and quality of the obtained extracts. These are the main reasons for recent shift in focus towards research, development and use of alternative extraction techniques. Extraction and BAC determination in such complex organic mixtures, by the use of conventional procedures, prescribed by Pharmacopoeia, very often requires longer period for sample preparation prior the analysis, and the use of various toxic organic solvents is needed, that could remain as trace element even in the final product, having a negative impact on the quality and the value of the product. Additional problems arising in traditional procedures of solid-liquid extraction are low quality of obtained extracts, slow mass transfer and low economic validity (high extraction price). All of these obstacles can be overcome only by the innovative approach and novel extraction techniques as an excellent solution for this challenge.

Under-utilized medicinal plants, food industry by-products and agricultural waste are considered as the promising source of valuable BACs (polyphenols, essential oil, carotenoids, lipids, etc.) with high potential for further application (Duenas and Garciá-Estévez, 2020). Since BACs present in natural resources could have many variations in molecular size, structure, stability and polarity, the selection

of an appropriate extraction procedure is a challenging step. Majority of novel approaches can be proved efficient in addressing these challenges and they certainly bring significant improvements comparing to traditional procedures, providing the extracts with high yield of target compounds and preserving the "green" nature of the process (Galanakis, 2012). The application of novel extraction techniques for valorization of under-utilized streams and production of new formulations with high potential for utilization in pharmaceutical, cosmetic and food sector are still very limited.

Considering the recent developments in food science and agriculture and the emerging demand toward the natural BACs, the aim of this manuscript is to provide a systematic review of the under-utilized medicinal plants, food industry by-products and agricultural waste based on their availability, sustainability, chemical profile and valorization potential (**Figure 1**).

< Please insert Figure 1 here >

Figure 1. Sustainable raw materials for efficient valorization and recovery of bioactive compounds

3. Under-utilized medicinal plants: Case study in Serbia

Despite the escalation in the production of synthetic drugs over the last couple of decades, medicinal plants still play a vital role in the life of rural communities (Manzoor Rashid et al., 2014). Moreover, there is an increased interest in cultivated and wild medicinal plants all over the world. Production of herbal medicaments, herbal cosmetic and dietetic products has become very popular. Increased use of traditional medicine, local plants and plant products in Europe is based on ethnobotanical studies and approved by modern science. Furthermore, discovering biological potential of some less known indigenous plant drug is encouraged as well (Pardo-de-Santayana et al., 2015).

When Slavs inhabited the Balkans, they brought their own primitive medicine from their ancient homeland, which was analogous to the medicine of the rest of the primitive European people (Grmek, 1959). During the Middle Ages, Serbian medicine developed under the influence of Byzantine and West European medicine using plants found in Europe, Asia and Africa. Throughout the period of Turkish occupation, Serbian people developed their folk medicine using local herbal drugs for the prevention and treatment of a wide range of illnesses (Jarić et al., 2011). In the early 19^{th} century, modern Serbian state began to develop and together with it the rural economy, as well. Medicinal and aromatic plants played an important role at this stage (Stevanović et al., 2014). By the early 1990s, Yugoslavia had been the largest exporter of medicinal and aromatic plants to the European market. However, after the breakup of Yugoslavia, its place was taken over by the surrounding countries such as Bulgaria, Poland and Hungary, which had similar climatic conditions (Filipović and Popović, 2014).

During the last two decades, there are between 1000 and 2000 ha per year under cultivation of medicinal plants in Serbia (Filipović and Popović, 2014; Maletić et al., 2010; Pljevljakuši´c et al., 2020; Turudija-Zivanovic and Zivanovic, 2012). **Table 1** shows an estimation of areas under medicinal plants cultivation in 2013 (Turudija Živanović, 2015) in comparison with 2021.

Table 1. Estimation of areas under medicinal plants in Serbia

No	Plant species	Estimation by S. Turudija Živanović	Estimation by M. Aćimović for 2021	Herbal drug

		for 2013 (Turudija Živanović, 2015)	(Aćimović et al., 2021)	
1	*Mentha piperita*	300	345	leaf
2	*Matricaria chamomilla*	250	273	flower
3	*Helichrysum italicum*	40	216	flower
4	*Petroselinum sativum*	200	215	leaf, seed, root
5	*Rosa canina*	-	157	fruit
6	*Melissa officinalis*	50	149	leaf
7	*Lavandula* sp.	40	133	flower
8	*Anethum graveolens*	-	47	leaf, seed
9	*Foeniculum vulgare*	60	39	fruit
10	*Hyssopus officinalis*	-	28	herb
11	*Anthemis nobilis*	-	25	flower
12	*Althaea officinalis*	50	24	leaf, flower, root
13	*Origanum* sp.	-	23	herb
14	*Rosa damascena*	-	20	flower
15	*Valeriana officinalis*	-	17	root
16	*Thymus vulgaris*	10	16	herb
17	*Satureja montana*	-	16	herb
18	*Angelica archangelica*	-	14	root, seed
19	*Coriandrum sativum, Carum carvi*	20	13	seed
20	*Ocimum basilicum*	5	10	herb
21	*Echinacea* sp.	-	6	herb, root
22	*Salvia officinalis*	10	3	leaf
23	*Calendula officinalis*	20	3	flower
24	Other	50*	34**	-
	Total	1095	1826	-

*Other (no specified)

**Other (*Achillea millefolium*, *Artemisia* sp., *Cynara scolymus*, *Geranium* sp., *Humulus lupulus*, *Iris* sp., *Juniperus communis*, *Nigella sativa*, *Trigonella foenum-graecum*)

The increase in areas under medicinal plants cultivation (from 1095 ha in 2013 to 1826 ha in 2021) is a consequence of the increase in commercial demand, especially requirements of food, pharmaceutical and cosmetic industries for constant supply of raw material in terms of quality and quantity (Basso et al., 1998). In the first place, lavender and immortelle (growth from 40 ha to 133 and 216 ha, respectively), which are used exclusively for essential oil distillation. Requirements for lemon balm are similar (growth from 50 ha in 2013 to 149 ha in 2021). Peppermint, chamomile, and parsley are three plants which are typically grown in Serbia, primarily as raw material for herbal tea production (peppermint and chamomile) and as spice (parsley). Plants that show commercial potential are rosehip and Damask rose, as well as dill, hyssop, roman chamomile, oregano, valerian, thyme, winter savory, angelica, basil and echinacea. In 2021, areas under fennel, coriander, caraway, marshmallow, sage and marigold decreased in comparison with 2013. This could be attributed to a high amount of manual work included in growing practices, harvest, and postharvest processing

(marshmallow and marigold), inappropriate quality (sage, fennel), or global climate changes, which significantly decrease yield and quality (coriander, caraway).

However, not every medicinal plant on the market is suitable for cultivation (Pljevljakuši´c et al., 2020). Collecting and processing medicinal plants are developed in hilly-mountainous areas of Serbia (Turudija-Zivanovic and Zivanovic, 2012). Serbia has a rich history and tradition of primary healthcare in a local communities, especially in Central (Kopaonik Mt), Eastern (Rtanj Mt, Timok and Negotin krajna), South-Eastern (Suva planina Mt, Vidlič Mt, Jablanica Mt, Svrljig Mt and Pčinja district,) and South-Western (Zlatibor Mt, Pešter Plateau and Sandžak) parts (Jarić et al., 2007; Marković et al., 2019; Matejić et al., 2020; Pieroni et al., 2011; Šavikin et al., 2013; Živković et al., 2021, 2020; Zlatković et al., 2014). Wild grown medicinal plants, traditionally used in Serbia are presented in **Table 2**. Apart from this, some plants are cultivated in gardens and yards, or collected from nature even if they are cultivated for medicinal purposes (listed in **Table 2**), while others are common vegetables (pepper, artichoke, parsnip, garlic, horseradish, common bean, etc.) or fruits (fig, quince, cherry, apricot, plum, grape, etc.), as well as ornamental plants (lily of the valley, majestic lily, wild privet, etc.).

Table 2. Wild grown medicinal plants traditionally used in Serbia

No	Plant species	Region	Herbal drug	Bioactive compounds	Reference
1	*Achillea millefolium*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	herb	Essential oil (0.4-0.8%) Phenolic compounds: acids (chlorogenic and dicaffeoylquinic acid isomers), flavones (luteolin and apigenin), and their corresponding glucosides	(Ivanović et al., 2022; Pljevljakušić et al., 2017)
2	*Agrimonia eupatoria*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Vidlič (Marković et al., 2019)	herb	Tannins (3-11%): ellagitannin and procyanidins Phenolic compounds: polyphenols (flavan-3-ols), flavonols (kaempferol and quercetin derivatives), flavones and phenolic acids. Essential oil (0.1-1.5%)	(Correia et al., 2006; Navaei and Mirza, 2013; Smeriglio et al., 2017)
3	*Allium ursinum*	Jablanica (Živković et al., 2021) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	fresh leaves	Organosulfur compounds (52.1-68.7%) Vitamin C	(Ivanova et al., 2009; Voća et al., 2021)
4	*Arctinium lappa*	Kopaonik (Jarić et al., 2007) Rtanj (Zlatković et al., 2014)	root	Phenolic compounds: acids (chlorogenic, caffeic and gallic acid), flavonoids	(Petkova et al., 2022)

		Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)			
5	*Arctostaphylos uva-ursi*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Timok (Matejić et al., 2020) Zlatibor (Šavikin et al., 2013)	leaf	Phenolic compounds (especially arbutin)	(Elwan et al., 2022)
6	*Artemisia absinthium*	Jablanica (Živković et al., 2021) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020)	herb	Essential oils Phenolic compouds: acids (coumaric, syringic, salicylic, chlorogenic and vanillic acids) Absinthin (sesquiterpene lactone) up to 0.2%	(Batiha et al., 2020)
7	*Asarum europaeum*	Jablanica (Živković et al., 2021) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)	root	Essential oil (0.7–4.1%)	(Akhlaq et al., 2022)
8	*Betula pendula*	Jablanica (Živković et al., 2021) Negotin (Janaćković et al., 2019) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	leaf	Phenolic compounds: acids (caffeine and chlorogenic acid, etc.), flavonol glycosides (hyperosids and quercetin, kaempferol and myricetin glycosides) Lignans Saponosids	(Vladimirov et al., 2019)
9	*Capsella*	Jablanica (Živković et al., 2021)	herb	Amino acids	(Grosso et al., 2011;

	bursa-pastoris	Kopaonik (Jarić et al., 2007) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)		Flavonoids (glycosides of quercetin, chrysoeriol, kaempferol, and isorhamnetin) Sterols	Kubínová et al., 2013; Ma et al., 2016)
10	*Chelidonium majus*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	herb	Alkaloids (chelidonine, chelerythrine and sanguinarine)	(Yao et al., 2011)
11	*Cichorium intybus*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	root	Phenolic compounds: acids (chlorogenic, isochlorogenic, neochlorogenic, caffeic and cichoric acids), glycosides (intybin) Polysaccharide: inulin (40–60%)	(Janda et al., 2021)
12	*Crataegus* sp.	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014)	leaf and flower	Flavonoids (rutin, apigenin, myricetin, quercetin, naringenin and kaempferol)	(Keser et al., 2014)

		Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)			
13	*Epilobium parviflorum*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Suva planina (Jarić et al., 2015) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)	herb	Phenolic compounds: acids (ellagic, chlorogenic, gallic, cinnamic, protocatechuic, caffeic and ferulic acid) Flavonoids (kaempferol, quercetin, myricetin, etc) Tannins (oenothein B, oenothein A, etc) Steroids (cholesterol, campesterol, stigmasterol, β-sitosterol, ursolic acid, corosolic acid, oleanolic acid)	(Merighi et al., 2021)
14	*Equisetum arvense*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	herb	Alkaloids (equisetin, nicotine, palustrine, and palustrinine) Flavonoids (apigenin, luteolin, kaempferol and quercetin glucosides) Saponosides Phytosterols (isobauerenol, epicholestanol, cholesterol, sitosterol, etc.)	(Boeing et al., 2021)
15	*Galium verum*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)	herb	Iridoid glycosides (secogalioside) Phenolic compounds: acids (chlorogenic acid and rutin) Phytosterols (sitosterol or campesterol) Triterpenes and anthraquinones	(Tomovic et al., 2021)
16	*Glechoma hederacea*	Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Svrljig (Matejić et al., 2020)	herb	Phenolic compounds: acids (rosmarinic, chlorogenic, caffeic acid, etc.) Flavonoids Terpenoids (such as ursolic and oleanolic acids)	(Zhou et al., 2021)

		Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)		Alkaloids (hederacins) Steroids	
17	*Hedera helix*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)	leaf	Phenolic compounds: flavonoids, anthocyanins, coumarins and phenolic acids Saponins (hederasaponin)	(Suica-Bunghez et al., 2020; Zdarta et al., 2019)
18	*Hypericum perforatum*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	herb	Phenolic compounds: acids (chlorogenic and coumaroylquinic acid) Flavonoids (hyperoside, rutin, quercitrin and kaempferol 3-o-rutinoside, etc) Hypericins and hyperforins	(Tusevski et al., 2019)
19	*Inula helenium*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Zlatibor (Šavikin et al., 2013)	root	Phenolic compounds: acids (gallic, 2-hydroxy benzoic, chlorogenic, neochlorogenic, caffeic, p-coumaric, sinapic, ferulic, vanillic, cinnamic acid) Flavonoids (quercetin, kaempferol, myricetin, catechin, epichatechin, etc) Polysaccharide (inulin)	(Buza et al., n.d.)
20	*Juniperus communis*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007)	fruit	Phenolic compounds: acids (hydroxybenzoic and hydroxycinamic acid)	(Gonçalves et al., 2022)

		Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)		Tanins (proantocyanidins) Flavonoids: biflavonoids (amento-flavone), flavones (apigenin), flavonols (quercetin, isoquercetin) Essential oil	
21	*Pinus sylvestris*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020)	resin (turpentine)	Phenolic compounds: acids (gallic, caffeic, syringic, p-coumaric, etc) Flavonoids: rutin, quercetin, apigenin etc. Essential oil	(Dziedziński et al., 2021)
22	*Plantago major*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	leaf	Flavonoids Alkaloids (indicain and plantagonin) Terpenoids Phenolic compounds: acids (caffeic acid derivatives) Iridoid glycosides (aucubin) Vitamins (provitamin A and vitamin C)	(Adom et al., 2017; Samuelsen, 2000)
23	*Polygonum aviculare*	Jablanica (Živković et al., 2021) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)	herb	Flavonoids (myricetin, quercetin, kaempferol, myricitrin, quercitrin, avicularin, juglanin, etc) Phenolic compounds: acids (gallic acid) Alkaloids (leonurine, gnoscopine, 8-acetyldolaconine) Sterols (sitosterol) Quinones	(Shen et al., 2018)
24	*Primula veris*	Jablanica (Živković et al., 2021) Pčinja (Živković et al., 2020)	root	Saponins (primulasaponin) Flavonoids (isorhamnetin-3-O-glucoside, astragalin and	(Tarapatskyy et al., 2021)

		Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)		catechin) Phenolic compounds: glycosides (primverin and primulaverin)	
25	*Pulmonaria officinalis*	Jablanica (Živković et al., 2021) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Vidlič (Marković et al., 2019)	herb	Anthocyanins Alkaloids (intermedine, lycopsamine, etc) Phenolic compounds (flavanone, flavonols, flavones, polyphenolic acids) Lignans	(Chauhan et al., 2022)
26	*Rosmarinus officinalis*	Jablanica (Živković et al., 2021) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020)	leaf	Flavonoids (genkwanin, cirsimaritin or homoplantaginin) Diterpenes (carnosic acid, carnosol or rosmanol) Triterpenoids (ursolic acid) Essential oil	(Borrás-Linares et al., 2014; Micić et al., 2021)
27	*Salix* sp.	Jablanica (Živković et al., 2021) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)	bark	Glucosides (salicin) Phenolic compounds: acids (caffeoylhexose and derivatives), flavanols and procyanidins (catechin, epicatechin, etc), flavonols (quercetin and derivatives) Lignans (sisymbrifolin) Stilbenes Tannins	(Piątczak et al., 2020; Warmiński et al., 2021)
28	*Sambucus nigra*	Kopaonik (Jarić et al., 2007) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)	flowers, fruits	Berries contain sugar, dietary fibre, protein and amino acids, cyanogenic glycosides (sambunigrin and prunasin) Flowers contain phenolic compounds (acids, flavonols)	(Młynarczyk et al., 2018)

		Zlatibor (Šavikin et al., 2013)			
29	*Sempervivum tectorum*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Zlatibor (Šavikin et al., 2013)	leaf	Phenolic compounds (kaempferol and quercetin derivatives) Volatile compounds	(Uzun et al., 2016)
30	*Solidago virgaurea*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Vidlič (Marković et al., 2019)	herb	Flavonoids (caffeoylquinic and salicylic acid derivatives) Saponines (virgaureasaponin) Essential oil	(Toiu et al., 2019)
31	*Symphytum officinale*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	root	Allantoin Phenolic compounds: acids (rosmarinic, *p*-hydroxybenzoic, caffeic, chlorogenic and *p*-coumaric acids)	(Salehi et al., 2019)
32	*Taraxacum officinale*	Jablanica (Živković et al., 2021) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020)	root, herb	Root contain: polysacharide (inulin), carotenoids (lutein), fatty acids, minerals, sugars, vitamins, mucilage and pectin. Herb contain: minerals, vitamins, fiber, and essential fatty acids, phenolic compounds	(Stanković et al., 2022; Wirngo et al., 2016)

		Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)			
33	*Teucrium chamaedrys*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Pčinja (Živković et al., 2020) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	herb	Flavonoids: flavone (luteolin) and three flavonoid glycosides (isoquercitrin, rutin and quercitrin) Phenolic compounds: acid and their derivatives derivatives (chlorogenic, *p*-coumaric and gentisic acids), Volatile compounds Iridoids	(Abdullah et al., 2022; Toiu et al., 2019)
34	*Teucrium montanum*	Kopaonik (Jarić et al., 2007) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	herb	Flavonoids (chicory acid and rutin) Phenolic compounds: acid and their derivatives (gallic, protocatechuic, gentisic, vanillic and syringic acid, chlorogenic, caffeic, coumaric, ferulic and 3,5-dimethoxy-4-hydroxycinnamic acid Volatile compounds	(Čanadanović-Brunet et al., 2006; Seremet et al., 2021)
35	*Tilia* sp.	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	flower	Flavonoids: glycosides of kaempferol (astragalin, tiliroside) and quercetin (isoquercitrin, rutin, hyperosid) Essential oil Phenolic compounds: acids (p-coumaric acid, caffeic acid) Vitamins: tocopherol Alkaloids (tiliine A and B, tiliamine A and B, tilacetine A and B) Carbohydrates Saponins	(Hake et al., 2022)
36	*Tussilago farfara*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007)	root, flower, leaf	Phenolic compounds: acids (caffeic acid, ferulic) Organic acids (ascorbic, malic and citric acids)	(Stoyanova and Perifanova-Nemska,

		Pčinja (Živković et al., 2020) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)		Amino acids (phenylalanine, valine, threonine, leucine, isoleucine and lysine)	2021)
37	*Urtica dioica*	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Negotin (Janaćković et al., 2019) Pčinja (Živković et al., 2020) Rtanj (Zlatković et al., 2014) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	herb, root, seed	Vitamins A, B and C Minerals (iron, potassium, calcium, magnesium) Phenolic compounds: acids and flavonoids Pigments (chlorophyll and carotenoids)	(Repajić et al., 2021)
38	*Verbascum* sp.	Jablanica (Živković et al., 2021) Kopaonik (Jarić et al., 2007) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019) Zlatibor (Šavikin et al., 2013)	flower	Phenolic compounds: acids (caffeic, chlorogenic, *p*-coumaric acid) Flavonoids (apigenin, rutin) Saponin glycosides (ilwensisaponin A and C) Iridoid glycosides (ajugol and picroside IV)	(El Gizawy et al., 2019)
39	*Viola odorata*	Jablanica (Živković et al., 2021) Suva planina (Jarić et al., 2015) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)	leaf, flower, root	Flavonoids (5,7-Dihydroxy-3,6-dimethoxyflavone, 5,7,4'-trihydroxy-3',5'dimethoxyflavone and 5,7,4'-trihydroxy-3'-methoxyflavone) Tannins Alkaloid (violin) Phenolic compounds Essential oils	(Karim et al., 2018; Shanker Gautam and Kumar, n.d.)
40	*Viola tricolor*	Jablanica (Živković et al., 2021)	herb	Phenolic compounds: acids (caffeic, p-coumaric, gentisic,	(Hellinger et al.,

		Kopaonik (Jarić et al., 2007) Svrljig (Matejić et al., 2020) Timok (Matejić et al., 2020) Vidlič (Marković et al., 2019)		protocatechuic, phydroxybenzoic, p-hydroxyphenylacetic, and vanillic acids) Flavonoids (violanthin, rutin) Polysaccharides (10% mucilage) Cumarins (umbelliferone and xanthine derivatives) Cyclotides	2014; Vukics et al., 2008)

One of the most important aspects in cultivation is the selection of the appropriate genotype, variety or hybrid suitable for certain agro-ecological conditions (Basso et al., 1998). However, there are limiting factors for growing medicinal plants in different regions of Serbia, such as hydro-thermic conditions as well as soil quality (available nitrogen, heavy metals, soil acidity etc.) (Obratov-Petković et al., 2006). Furthermore, in order to produce high-quality herbal products, from cultivated as well as wild plants, attention must be paid to phytochemical variations due to weather conditions, cultivation parameters, harvest time and technique (Aćimović et al., 2022, 2021; Tanko et al., 2005). Postharvest processing, storage and extraction are also critical factors for quality (Pandey and Pandey, 2017). Accoridng to broad diversity and unique chemical profile of widely grown medicinal plants, it could be concluded that they should be thoroughly explored as raw material for isolation of various BACs in order to obtained novel extracts for high-value applications.

4. Cakes obtained from oilseeds

The cultivation of oilseeds shows rising trend worldwide (**Figure 2**). The top five oilseeds (soybeans, cottonseed, rapeseed, peanuts and sunflower) in terms of annual crop yield accounted cumulatively for $600x10^6$ tons of seeds during 2020 season. Large portion of cultivated oilseeds is utilized as a raw material for oil pressing industry. Therefore, large amount of oil cakes is annually generated as a sidestream from oil pressing industry.

< Please insert Figure 2 here >

Figure 2. Worldwide production of the top five oilseeds in the period from 2016 to 2020 (Http://www.fao.org/faostat/en/#data, 2022)

Besides oil cakes obtained from these five oilseeds, there are many other alternative oil cakes/pomaces from plants such as sesame (Nascimento et al., 2012), blackberry, sour cherry, linseed, pumpkin, grape (Krulj et al., 2021), raspberry (Li et al., 2022), olive (Antónia Nunes et al., 2018), plum (Čakarević et al., 2019b), apricot (Čakarević et al., 2019a), hemp (Serrapica et al., 2019), corn (Li et al., 2018), walnut (Garcia-Mendoza et al., 2021) etc. Some of these plant materials such as olive and sesame are commonly used for oil production while apricot, plum, pepper, blackberry, raspberry seeds/kernels are underutilized and could be further valorized by food industry as a source of edible oils and later as a source of oil cakes. In the **Table 3,** chemical composition of oilseeds, crops, nuts, fruits and vegetables are presented.

Table 3. Proximate composition of oil cakes obtained from different plants

Plant	Plant part	Ash (g/100g)	Crude protein (g/100g)	Crude fat (g/100g)	Total dietary fiber (g/100g)	Total carbohydrate (g/100g)	Moisture (g/100g)	Extraction technique	Reference
Almond	-	5.30±0.20	51.30±0.90	19.0±0.50	-	18.80±0.60	5.80±0.20	-	(Montrimaitė and Moščenkova, 2018)
Apricot	kernel	-	42.31±0.53	22.04±0.23	8.94±0.40	15.07±0.01	8.90±0.10	CP	(Čakarević et al., 2019a)
Apricot	kernel	-	43.43±0.80	6.37±0.54	8.06±0.19	23.23±0.06	6.25±0.03	SFE	(Čakarević et al., 2019a)
Blackberry*	seed	1.85±0.08	13.20±1.02	6.15±0.08	62.09±0.95	-	3.98±0.00	CP	(Krulj et al., 2021)
Blue poppy*	seed	0.91±0.15	32.28±1.33	14.42±0.47	35.76±1.60	-	7.44±0.00	CP	(Krulj et al., 2021)
Corn	germ	1.85	21.53	2.07	53.45	-	7.71	-	(Li et al., 2018)
Cottonseed	seed	6.5	34.7	8.8	18.0	-	7.2	-	(AANYU and ONDHORO, 2016)
Flaxseed*	seed	4.99±0.12	35.41±1.65	13.52±0.54	37.25±1.78	-	8.68±0.04	CP	(Krulj et al., 2021)
Groundnuts*	-	6.72±0.04	42.70±0.50	9.00±0.2	39.40±0.01	-	-	CP	(Behera et al., 2013)
Hazelnut	-	5.30±0.20	35.70±0.80	23.40±0.40	-	27.50±0.70	8.10±0.35	-	(Montrimaitė and Moščenkova, 2018)
Hemp	seeds	7.07±0.90	28.97±3.76	10.20±3.49	-	-	8.13±0.50	-	(Serrapica et al., 2019)

Muscat* pumpkin	hull and kernel	6.59±0.32	41.61±1.26	14.94±0.40	23.46±1.2	-	5.85±0.04	CP	(Krulj et al., 2021)
Olive*	-	1.90±0.00	7.40±0.20	5.80±0.30	-	84.90±0.40	61.10±0.70	-	(Antónia Nunes et al., 2018)
Palm	kernel	6.10±1.20	17.60±1.40	5.50±0.30	11.50±1.30	50.40±2.30	8.90±1.10	-	(Zarei et al., 2012)
Pepper	seed	3.6±0.05	17.48±0.46	7.38±0.38	-	-	8.79±0.27	E and CP	(Yilmaz et al., 2015)
Pepper	seed	4.82±0.17	18.76±0.71	8.15±0.75	-	-	8.49±0.53	R and CP	(Yilmaz et al., 2015)
Pepper	seed	3.84±0.17	17.08±0.94	9.85±0.00	-	-	8.98±0.25	CP	(Yilmaz et al., 2015)
Plum	kernel	-	50.69±0.53	10.09±0.17	5.86±0.32	12.77±0.02	10.80±0.15	CP	(Čakarević et al., 2019b)
Plum	kernel	-	48.93±0.63	10.63±0.12	11.57±0.35	12.59±0.05	6.27±0.32	SFE	(Čakarević et al., 2019b)
Pomegranate	seeds	4.10±0.22	14.91±0.35	0.95±0.24	-	-	8.45±0.01	-	(Serrapica et al., 2019)
Pumpkin*	kernel	6.95±0.18	50.96±2.41	18.10±0.63	13.73±0.86	-	7.69±0.05	CP	(Krulj et al., 2021)
Rapeseed	seed	7.30±0.00	40.60±0.00	2.80±0.10	-	-	8.00	CP	(Fetzer et al., 2018)
Raspberry*	seed	1.75±0.06	10.55±0.86	5.73±0.20	71.65±1.74	-	3.26±0.01	CP	(Krulj et al., 2021)
Red grape*	seed	2.51±0.11	12.79±0.87	6.50±0.23	74.61±3.50	-	8.87±0.03	CP	(Krulj et al., 2021)

Sesame*	seed	6.09±0.34	47.15±2.06	16.96±0.66	15.48±0.54	-	4.82±0.02	CP	(Krulj et al., 2021)
Sour cherry*	shell and kernel	0.96±0.15	6.14±0.32	5.98±0.32	79.73±4.20	-	3.51±0.03	CP	(Krulj et al., 2021)
Soybean*	-	7.48±0.06	50.50±0.60	2.10±0.30	40.20±0.01	-	-	CP	(Behera et al., 2013)
Sunflower	-	6.15±0.04	21.60±1.87	14.16±0.04	12.64±0.05	36.52±1.11	8.93±0.11	CP	(Petraru et al., 2021)
Walnut	kernel	5	27	37	-	28	3	MP	(Garcia-Mendoza et al., 2021)

* expressed on dry matter; CP- cold pressing; SFE-supercritical fluid extraction; MP-mechanical pressing; E-enzymatic pre-treatment; R-roasting pre-treatment.

The chemical composition of oil cakes depends on many factors which could be grouped as factors related to raw material or oil extraction technology. The quality of raw material which is submitted to oil extraction is mainly determined by plant species, growing climate conditions, and application of various agro-technical methods etc. The impact of plant material on oil cakes chemical composition could be easily understood, considering the following examples: proximate composition of blackberry, raspberry, sour cherry or red grape oil cakes which are rich in fiber fraction (62.09-79.73 g/100g) while pumpkin, plum, soybean or almond meals (50.50-51.30 g/100g) are rich in plant proteins (**Table 3**). Apart from the main oil extraction process, pre-treatment technology of plant material could also significantly alter the chemical composition of residual cakes. For example dehulling which could be applied as pre-treatment before oil extraction from pumpkin seeds, flaxseed, sunflower etc. will result in complete or partial separation of outer seed layers that are rich in fibers from central parts of seeds that are oil-high and protein-high fraction (Krulj et al., 2021). The effectiveness of impurities removal would also significantly determine composition of meals since chemical composition of impurities (even other types of edible seeds) could vary to a great extent. Enzymatic pre-treatment and particularly heating pre-treatment could be applied mainly to increase oil extraction yield which will later lead to production of oil cakes with lower residual oil content (g/100g cake) but higher content of proteins, fibers, ash (g/100g cake) etc. Yılmaz et al. (Yilmaz et al., 2015) reported that residual oil content was 7.38 g/100g, 8.15 g/100g and 9.85 g/100g in pepper meals pre-treated with hemicelluloses and protease, pre-treated with roasting at 150 °C for 25 min, and without any pre-treatment, respectively. Conventional oil extraction is mainly performed by mechanical pressing with screw/expeller press, hydraulic press or by solvent extraction (Kumar et al., 2017). Chemical composition of oil cakes would also greatly depend on applied oil recovery technology since oil cakes after mechanical pressing will have higher crude fat content compared to meals obtained with oil solvent extraction. Furthermore, a recent study reported that crude fat content in apricot meal was 6.37 g/100g and 22.04 g/100g after supercritical fluid extraction (SFE) and cold pressing, respectively (Čakarević et al., 2019a). However, it is noteworthy that solvent extraction of fatty acid oils on industrial scale is commonly performed with hexane rather than with SFE technology.

Due to high diversity of oil cakes chemical composition, oil cakes could be directly or indirectly utilized for wide a spectrum of applications in the food industry (**Figure 3**).

< Please insert Figure 3 here >

Figure 3. Direct and indirect applications of oil cakes in the food industry

Some intuitive and direct applications could be incorporation of highly nutritive (high protein content, high content of essential amino acids etc.) oil cakes in food products such as corn base snacks (Nascimento et al., 2012), fortified breads (Tarek-Tilistyák et al., 2014), gluten-free bread sticks (Caponio et al., 2022), gluten-free batter (Burbano et al., 2022), oil cake based spread (Radočaj et al., 2012), yoghurt-like beverages (Gul et al., 2022) etc. Dietary fibers which are capable to combat diabetes, high blood cholesterol and may aid in colon cancer prevention could be added in functional products via oil cakes with high content of dietary fibers such are raspberry and strawberry meals (Kowalczewski et al., 2019). These high protein or high fiber cakes could be also used as animal feed (Haese et al., 2022; Hakim et al., 2022) which are later utilized to produce animal based food products. Development of sustainable, recyclable or even edible packaging materials is another food

industry problem which could be partly tackled using residual oil cakes as a raw material for bioplastics and biofilms. Moreover, a recent study reported that pumpkin oil cake pouches could be used as a high oxygen-barrier, antioxidant and edible packaging material, for preventing the oxidation of flaxseed oil (Hromiš et al., 2022). Similar protein based films could also be made of soybean, rapeseed, sunflower, cottonseed, groundnut, sesame, black cumin, hemp, and cardoon meals (Mirpoor et al., 2021). Edible and non-edible oil cakes are used for production of biodiesel and biogas (Mohanty et al., 2022) which could be further utilized to produce bioelectricity. In that regard, part of the fossil based energy necessary for food industry could be substituted with sustainable and green bioenergy from oil cakes. Another indirect application of oil cakes in food industry could be substitution of chemical fertilizers with biofertilizers (Datta et al., 2017). Non-edible cake from castor, karanja, neem and mustard can be used as biofertilizers due to their high content of nitrogen, phosphorus and potassium (Datta et al., 2017) which are necessary nutrients for plants growth.

Certain oil cakes contain not only proteins, fats, carbohydrates, minerals and other essential nutrients required for human body, but also plant secondary metabolites such as polyphenols, tocopherols, carotenoids, terpenoids, phytosterols etc. Thus, oil cakes are also used as a raw material for the production of various nutraceuticals and pharmaceuticals. In the **Table 4,** the oil cakes which could be used as an alternative source of various secondary metabolites are presented.

Table 4. Semi-defatted/defatted oil cakes and extraction techniques used for recovery of plant secondary metabolites

Plant	Plant material	Secondary metabolite	Extraction technique and solvent	Reference
pistachio	semi-defatted nuts	phenolic acids, flavonols, flavanones, flavanols, and isoflavones	SBE[1] with water	(Bodoira et al., 2020)
chia	semi-defatted seeds	polyphenols	SLE[2] with acetone-water	(Aranibar et al., 2021)
flaxseed	semi-defatted seeds	polyphenols, tocopherols and phytosterols	SLE with hexane	(Zhang et al., 2021)
grape	defatted seeds	polyphenols	SLE with methanol-water	(Mora-Garrido et al., 2022)
hemp	defatted seeds	proanthocyanidins and cannabinoids	EAE[3] with water	(Pap et al., 2020)
hemp	semi-defatted seeds	terpenoids, carotenoids and cannabinoids	SLE with methanol-chloroform	(Banskota et al., 2022)
jojoba	defatted seeds	polyphenols and simmondsins	MAE[4] with ethanol-water	(Feki et al., 2021)
olive	defatted pomace	secoiridoids, phenolic alcohols, flavonoids, lignans	PLE[5] with ethanol-water	(Pavez et al., 2019)

olive	pâte	phenolic acids, flavonoids and phenylethanoids	UAE[6] with methanol-water	(Peršurić et al., 2020)
palm	semi-defatted kernels	phenols and squalene	SE[7] with isopropanol	(Nuchdang et al., 2022)
rapeseed	semi-defatted seeds	tocopherols and carotenoids	UTA[8] with methanol-tetrahydrofuran	(Franke et al., 2010)
rapeseed	semi-defatted seeds	tocopherols and carotenoids	SFE[9] with carbon-dioxide	(Uquiche et al., 2012)
raspberry	defatted seeds	ellagic acid	SLE with NADES[11]	(Teslic et al., 2022)
safflower	semi-defatted seeds	tocopherols	UTA with methanol-tetrahydrofuran	(Franke et al., 2010)
sesame	defatted seeds	phenolic acids, flavonoids and lignans	SLE with methanol-water	(Mekky et al., 2019)
sesame	semi-defatted seeds	phenolic acids, flavones, flavonols, isoflavones and lignans	SBE with water	(Bodoira et al., 2020)
sesame	semi-defatted seeds	polyphenols and lignans	PEF-HVED-AE[10] with ethanol-water	(Sarkis et al., 2015)
sunflower	semi-defatted kernels and shells	chlorogenic acid	MAE with ethanol-water	(Náthia-Neves and Alonso, 2021)
sunflower	semi-defatted seeds	tocopherols	UTA with methanol-tetrahydrofuran	(Franke et al., 2010)
walnut	semi-defatted nuts	carotenoids	SFE with carbon-dioxide	(Bardeau et al., 2015)
wheat	defatted germ	polyphenols	MAE with ethanol-water	(Teslić et al., 2019)

[1] SBE – Subcritical extraction; [2] SLE – solid-liquid extraction; [3] EAE – enzyme-assisted extraction; [4] MAE – microwave-assisted extraction; [5] PLE – Pressurized liquid extraction; [6] UAE – ultrasound-assisted extraction; [7] SE – Soxhlet extraction; [8] UTA – Ultra-turrax-assisted extraction; [9] SFE – Supercritical fluid extraction; [10] PEF-HVED-AE – Pulsed electric field-high voltage electrical discharges-assisted extraction; [11] NADES – Natural deep eutectic solvents extraction.

Data from a thorough scientific literature review related to the use of semi-defatted and defatted cakes as a raw material for isolation of secondary metabolites revealed that the most of reports are either dealing with recovery of polar molecules (various phenolic like compounds) or extraction of less polar or non-polar compounds (tocopherols, carotenoids, phytosterols etc.). The former metabolites are in most of the cases extracted using water, methanol, ethanol, and various mixtures of aqueous methanolic or aqueous ethanolic solutions (**Table 4**). In certain cases, polyphenols are

extracted even with other organic solvents such as isopropanol and acetone (Aranibar et al., 2021; Nuchdang et al., 2022). Water and ethanol are particularly interesting as extraction medium for recovery of polar compounds from oil cakes since they are generally recognized as safe (GRAS) solvents and could be further utilized in some food products without additional purification steps which will decrease cost of production. In the last five years natural deep eutectic solvents (NADES) are gaining popularity among scientific community since NADES are composed only of edible, recyclable, less or non-toxic compounds which are predominantly present in nature (Teslić et al., 2022). More precisely, water, sugars, fatty acids, organic salts, organic acids, amino acids, terpenes, alcohols and other compounds that are commonly used by the food industry, may be mixed in the appropriate ratio to create solvents with tailored properties suitable for a various applications in the food industry. Since, the most of researched phenolics are flavonoids and phenolic acids (**Table 4**), which may interact to a certain degree with the plant cell wall components (e.g. pectin and hemicellulose) (Siemińska-Kuczer et al., 2022), recovery of polyphenols is often performed with some kind of assisted-extraction technique which will break bonding between cell wall compounds and polyphenols. Thus, often utilized extraction techniques are microwave-assisted extraction (MAE) (Teslić et al., 2019), ultrasound-assisted extraction (Peršurić et al., 2020), enzyme-assisted extraction (Pap et al., 2020), acidic-hydrolysis-assisted extraction (Teslic et al., 2022), pulsed-electric field extraction (Sarkis et al., 2015), subcritical water extraction (Bodoira et al., 2020) etc. One of the differences between these extraction techniques is the force or phenomenon which is causing damage to the plant cell wall. In MAE, it is microwave irradiation which causes heat damage against the cell wall, in UAE are cavitation bubbles which abrupt bursts induce shear stress to the wall, in EAE various enzymes (cellulase, hemicellulase, protease etc.) will cause detachment of polyphenols from other cell plant compounds. The lipophilic compounds (e.g. tocopherols and carotenoids) present in oil cakes are actually dissolved in oil phase thus their recovery is mainly possible from semi-defatted meals (**Table 4**). Since oil cakes obtained after mechanical pressing often contain both, lipophilic and hydrophilic plant secondary metabolites, semi-defatted oil cakes can be used in a sequel extraction process aiming to increase extraction efficiency and to increase exhaustion of plant material. Such process will enable recovery of hydrophobic extracts in the first step and in the second step isolation of hydrophilic extracts. The extraction of tocopherols, carotenoids and similar compounds is mainly performed with hexane or carbon-dioxide (CO_2). The latter is advantageous comparing to organic solvents since it is environmentally safe, cheap, non-toxic and non-flammable (Marić et al., 2020). The most commonly utilized extraction techniques are SLE, SFE and SE (**Table 4**). SLE and SE are considered as convectional extraction techniques which are disadvantageous comparing to supercritical CO_2 extraction (SCO_2E) since these techniques are performed with toxic organic solvents. In addition, they are less cost-efficient and time consuming. Often mentioned issue in literature with SCO_2E is high cost of required equipment which could be justified only if obtained extracts contain high concentration of valuable compounds.

5. Herbal dust from filter tea processing

Nowadays the focus of the global research lies in the elimination of negative impact on environment and in the increase of the resource utilization. The amount of waste from various industries is only increasing and its further processing is more than needed. Industrial waste could be classified into hazardous and nonhazardous waste. The first group includes radioactive waste and waste emitted from chemical processing or mining. On the other side, the nonhazardous waste includes for instance sugar milling, paper production or by-products generated from food processing units, such as herbal

dust which remains after filter-tea production (Kathir et al., 2022). Herbal dust from filter-tea industry belongs to bio-based waste and represents the main aim of this section, due to its numerous positive effects and benefits. Although the content of bioactive compounds in unprocessed herbal material is higher than in the processed one, herbal dust still represents a highly valuable source of polyphenols and lipophilic compounds (Mrkonjić et al., 2021b; Naffati et al., 2017a). Accordingly, its popularity in the last decades has been rising progressively. The reason behind refers to the huge processability of this fraction in terms of the possibility of obtaining various types of extracts or essential oils, and also in its rich chemical composition. This implies the potential use of processed herbal dust fraction in the food, cosmetic and pharmaceutical industries (Vidović et al., 2013). According to local filter-tea manufacturer data for the year 2021, total waste during *Thymus serpyllum* L. filter-tea production reached 33.33% (2500 kg of herbal dust was produced from 7500 kg plant material). Herbal dust from filter-tea industry represents a particular fraction whose mean particle size is smaller than the filter-tea bag pores (0.315 mm). Due to the impracticability of using herbal dust in filter-tea industry, this research focused on finding alternative ways for adequate valorization of this waste fraction. Even though the possibility of agglomeration and repackaging exists, this process proved to be inefficient in terms of techno-economical costs. The production of liquid and dry extracts, as well as essential oils, using this kind of starting material has proven to be an excellent approach in the valorization of such industrial waste. For that purpose, conventional and novel extraction and distillation techniques were developed.

In order to produce extracts and isolate polyphenols and essential oils from herbal dust, several approaches based on the mass transfer principles from the plant material to the liquid phase could be used. There are several conventional (maceration, percolation, distillation, Soxhlet extraction), as well as novel techniques, such as extractions assisted with ultrasounds and microwaves, SFE, subcritical water extraction (SWE), PLE, PEF, NADES extraction, enzyme digestion, etc. In comparison with novel extraction techniques, conventional solid-liquid extraction has certain disadvantages in terms of requiring longer operation time and resulting in lower extraction yields (Belwal et al., 2018). Better extraction efficiency and accelerated solvent penetration into the cellular material and increased mass transfer from solid to liquid phase could be achieved by application of external energy such as ultrasounds or microwaves, as well as by using NADES or solvents on sub- or supercritical level. The **Table 5** represents optimal extraction conditions, under which the best-quality extracts were obtained from various herbal dusts considering total phenols yield. As a solid phase for following extractions, herbal dusts of the following plants were used: *Thymus serpyllum* L., *Salvia officinalis* L., *Arctostaphylos uva ursi*, *Achillea millefolium*, *Rosa canina*, *Aronia melanocarpa* and wild apple.

Table 5. Optimal extraction parameters for utilization of herbal dusts of various plants

Herbal dust	Extraction technique	Process parameters	Extracted compounds	Total phenols yield	Reference
T. serpyllum L.	CE	Solvent: 60% ethanol Solid-liquid ratio: 1:20 (w/V) Extraction time: 24 h Shaking speed: 150 rpm	Flavanones, flavonols, flavones, isoflavones and phenolic acids	1.74 g GAE/100 g DW	(Mrkonjić et al., 2021b)
	UAE	Solvent: 45% ethanol	Flavan-3-ols,	4.39 g GAE/100	(Mrkonjić et al.,

		Solid-liquid ratio 1:20 (w/V) Extraction time: 70 min Ultrasound power: 42 W/L Temperature: 70.28 °C	flavanones, flavonols, flavones, coumarins, tannins and phenolic acids	g DW	2021b)
	MAE - RSM	Solvent: 52.19% ethanol Solid-liquid ratio 1:23.64 (w/V) Extraction time: 20 min Irradiation power: 400 W	Flavan-3-ols, flavanones, flavonols, flavones, cumarins, tannins and phenolic acids	4.86 g GAE/100 g DW	(Mrkonjić et al., 2022)
	MAE - ANN	Solvent: 45% ethanol Solid-liquid ratio 1:30 (w/V) Extraction time: 5 min Irradiation power: 400 W	Polyphenols	4.36 g GAE/100 g DW	(Mrkonjić et al., 2022)
	PLE	Solvent: 30% ethanol Solid-liquid ratio 1:20 (w/V) Extraction time: 30 min Temperature: 170 °C	Flavan-3-ols, flavanones, flavonols, flavones, isoflavones, tannins and phenolic acids	6.75 g GAE/100 g DW	(Mrkonjić et al., 2021a)
	NADES	Solvent: L-prolin:glycerine:H_2O (1:2:1) Solid-liquid ratio 1:28 (w/w) Extraction time: 180 min Temperature: 65 °C	Epicatechin, luteolin, quercetin, gallic, caffeic and rosmarinic acids	7.14 g GAE/100 g DW	(Pavlić et al., 2022)
S. officinalis L.	CE	Solvent: 60% ethanol Solid-liquid ratio: 1:20 (w/V) Extraction time: 24 h Shaking speed: 150 rpm	Polyphenols	4.69 g GAE/100 g DW	(Branimir Pavlić et al., 2016)
	UAE	Solvent: 60% ethanol Solid-liquid ratio 1:20 (w/V) Extraction time: 80 min Ultrasound power: 42.54 W/L Temperature: 75.40 °C	Caffeic, *p*-coumaric, ferulic and rosmarinic acid	9.88 g GAE/100 g DW	(Zeković et al., 2017)
	MAE	Solvent: 46.2% ethanol Solid-liquid ratio 1:40 (w/V) Extraction time: 18.7 min Irradiation power: 600 W	Caffeic, *p*-coumaric, ferulic and rosmarinic acid	10.38 g GAE/100 g DW	(Zeković et al., 2017)
	SFE	Pressure: 297.52 bar Temperature: 44.4 °C CO_2 flow rate: 0.4 kg/h Extraction time: 240 min	Terpenoids	-	(Pavlić et al., 2018)
	SWE	Solvent: double-distilled water	Polyphenolic and	7.98 g GAE/100	(Branimir Pavlić et

		Solid-liquid ratio 1:10 (w/V) Temperature: 201.5 °C Extraction time: 15.8 min HCl concentration: 0%	lipophilic compounds	g DW	al., 2016)
	HD	Solid-liquid ratio 1:20 (w/V) Distillation time: 120 min	Terpenoids	-	(Pavlić et al., 2017b; Radivojac et al., 2020)
	MWHD	Solid-liquid ratio 1:40 (w/V) Distillation time: 120 min Irradiation power: 800 W	Terpenoids	-	(Radivojac et al., 2020)
	SE	Solvent: methylene chloride and *n*-hexane	Terpenoids	-	(Pavlić et al., 2018)
A. uva ursi	CE	Solvent: 30% ethanol Solid-liquid ratio: 1:5 (w/V) Extraction time: 24 h Shaking speed: 150 rpm Temperature: 25 °C	Arbutin, rutin, hyperoside, isoquercitrin and gallic acid	7.29 g GAE/100 g DW	(Naffati et al., 2017a)
	UAE	Solvent: 70% ethanol Solid-liquid ratio 1:10 (w/V) Extraction time: 80 min Ultrasound power: 144 W Temperature: 80 °C	Arbutin, rutin, hyperoside, isoquercitrin and gallic acid	15.37 g GAE/100 g DW	(Živković et al., 2022)
	SWE	Solvent: double-distilled water Solid-liquid ratio: 1:10 (w/V) Extraction time: 10 min HCl concentration: 1.5% Temperature: 151.2 °C	Arbutin, rutin, hyperoside, isoquercitrin and gallic acid	9.84 g GAE/100 g DW	(Naffati et al., 2017a)
A. millefolium	CE	Solvent: 70% ethanol Solid-liquid ratio: 1:20 (w/V) Extraction time: 24 h Shaking speed: 150 rpm Temperature: 25 °C	Polyphenols	18.29 g GAE/100 g DW	(Milutinović et al., 2015)
	MAE	Solvent: 70% ethanol Solid-liquid ratio 1:40 (w/V) Extraction time: 33 s Irradiation power: 170 W	Polyphenols	23.77 g GAE/100 g DW	(Milutinović et al., 2015)
	SWE	Solvent: double-distilled water Solid-liquid ratio: 1:10 (w/V) Extraction time: 16.5 min HCl concentration: 0%	Polyphenols	11.80 g GAE/100 g DW	(Vladić et al., 2020)

		Temperature: 198 °C			
	SFE	Pressure: 300 bar Temperature: 60 °C CO_2 flow rate: 0.194 kg/h Extraction time: 300 min	Chlorophylls, carotenoids, hydrocarbons, tocopherols and phytosterols	-	(B. Pavlić et al., 2016a; Vidović et al., 2021)
	SE	Solvent: methylene chloride and *n*-hexane	Chlorophylls and carotenoids	-	(Pavlić et al., 2015)
R. canina	SFE	Pressure: 300 bar Temperature: 60 °C CO_2 flow rate: 0.194 kg/h Extraction time: 300 min	Chlorophylls, carotenoids, hydrocarbons, tocopherols and phytosterols	-	(B. Pavlić et al., 2016a; Vidović et al., 2021)
A. melanocarpa	CE	Solvent: 50% ethanol Solid-liquid ratio: 1:5 and 1:10 (w/V) Extraction time: 24 h Temperature: 25 °C	Phenols, flavonoids and anthocyanins	4.60 g GAE/100 g DW	(Gavaric et al., 2018; Ramić et al., 2015)
	UAE	Solvent: 50% ethanol Solid-liquid ratio 1:5 (w/V) Extraction time: 80.1 min Ultrasound power: 206.64 W Temperature: 70 °C	Phenols, flavonoids and anthocyanins	7.53 g GAE/100 g DW	(Ramić et al., 2015; Vidović et al., 2019)
	SWE	Solvent: double-distilled water Solid-liquid ratio: 1:10 (w/V) Extraction time: 15.2 min HCl concentration: 0% Temperature: 120.4 °C	Phenols, flavonoids and anthocyanins	3.29 g GAE/100 g DW	(Gavaric et al., 2018)
Wild apple	CE	Solvent: 50% ethanol Solid-liquid ratio: 1:10 (w/V) Extraction time: 24 h	Polyphenols	0.03 g GAE/100 g DW	(Naffati et al., 2017b)
	UAE	Solvent: 70% ethanol Solid-liquid ratio 1:10 (w/V) Extraction time: 40, 60 and 80 min Ultrasound power: 72, 144 and 216 W Temperature: 40, 60 and 80 °C	Polyphenols	0.06 g GAE/100 g DW	(Naffati et al., 2017b)
	MAE	Solvent: 40% ethanol Solid-liquid ratio: 1:10 (w/V) Extraction time: 15.2 min	Polyphenols	0.04 g GAE/100 g DW	(Pavlić et al., 2017a)

		Irradiation power: 400 W			

CE – conventional solid—liquid extraction; SE – Soxhlet extraction; HD – hydrodistillation; MWHD – microwave-assisted hydrodistillation; GAE – gallic acid equivalent; DW – dry weight.

According to the studies, the most frequently used extraction techniques are CE, UAE and MAE. Despite that, SWE, PLE, SFE and NADES extractions could be identified as extraction techniques with higher quality extracts in comparison with aforementioned, in terms of polyphenols yield and antioxidant activity. However, considering some disadvantages like initial investment, operational costs and in the case of NADES, difficulties with down-stream process and extract purification, MAE and UAE still present very good approach for isolation of bioactive compounds from different plant materials. It could also be noticed that only solvents that respect safe food production and the concept of "green chemistry" were used. The water-ethanol mixtures are desirable, due to their capability of extraction of different polar compounds. In addition, they are adjustable and flexible for safe human consumption. The use of CO_2 at supercritical, water on subcritical level, as well as NADES solvents, which are even more desirable from the green technologies' aspect, show direction in which investigation of novel technologies should be developed.

Extracts obtained from herbal dust in the form of essential oil, liquid or powder extract, showed antioxidant and antibacterial activity, which has opened new horizons in terms of potential applications in the agri-food sector, cosmetic and pharmaceutical industries as a substitution for synthetic products. The application perspective of aforementioned extracts as natural additives in food products such as meat and dairy products has been determined in the following studies. In order to find a way to enhance safety and stability, and to extend shelf life of fresh pork sausages, *S. officinalis* extract and essential oil were incorporated. Essential oil was obtained by hydrodistillation, while the extract was obtained by SFE. The effect on the pH value, sensory properties, oxidative and microbiological stability on fresh pork sausages was investigated and the result showed retarded microbial growth. This signifies successful application of essential oil and extracts from *S. officinalis* herbal dust as antimicrobial agents in fresh pork sausages (Šojić et al., 2018, 2017). Furthermore, essential oil and SFE extracts obtained from *S. officinalis* herbal dust were also investigated as a natural antimicrobial agent against *Listeria monocytogenes* (Danilović et al., 2021a) and *Escherichia coli* growth in minced pork (Danilović et al., 2021b). It was concluded that the number of *L. monocytogenes* could be significantly reduced in minced meat up to 6 days of refrigeration using concentrations of essential oil and supercritical extract of 0.150 and 0.300 µL/g, respectively (Danilović et al., 2021a). In addition, using 0.4 and 0.6 µL/g concentration of essential oil and 0.4, 0.6 and 1.0 µL/g concentration of SFE extract demonstrated reduction of the number of *E. coli* as well as shelf life extension of minced pork up to 8 days of refrigeration (Danilović et al., 2021b). Decreased discoloration, reduced protein and lipid oxidation, and inhibited microbial growth lead to shelf life extension of ground pork patties with incorporated *T. serpyllum* extracts. *T. serpyllum* lipid extracts used in this study were obtained using SFE and according to microbiological, physicochemical and sensory properties, they could serve as natural antioxidant in ground meat patties (Šojić et al., 2020). On the other side, Vukić et al. (Vukić et al., 2021) demonstrated successful production of kombucha fresh cheese with non-conventional starter culture, and addition of *S. officinalis* essential oil and incorporated SFE extract. Due to enhanced antioxidant activity and satisfied sensory characteristics of kombucha fresh cheese, prepared in the aforementioned way, *S. officinalis* herbal dust has great perspective in the field of innovative and valuable dairy products.

In comparison with unprocessed plant material that is mowed from the field, dried and stored under controlled conditions, herbal dust also presents the high-valuable source of polyphenols and antioxidants. Due to its rich and divergent polyphenols profile, and potential use in the form of liquid extract, powder or essential oil is becoming more frequent and diverse. So far, only several different plants have been investigated taking into account their herbal dust and its valorization. Based on previous research on *T. serpyllum*, *S. officinalis*, *A. uva ursi*, *A. millefolium*, *R. canina*, *A. melanocarpa* and wild apple, there are very good and efficient ways of utilization of the waste from filter-tea factories. It might be desirable to pay attention of other researchers and companies engaged in this field of study, to work on improving and developing waste recovery techniques by reducing production losses and increasing its efficiency and sustainability.

6. Agricultural waste

Agricultural wastes are non-product outputs obtained as a result of food production or processing of agricultural products which often contain compounds that can be useful to people, but their economic value is not sufficient to cover the costs of their further exploitation (Obi et al., 2016). Agricultural wastes are divided into two groups: the crop residue group, such as plant residues that are left on fields after crop harvest (*i.e.*, roots, leaves, straws, stubbles etc.), and agro-industrial residue group which includes by-products of post harvesting and food processing. It is estimated that the global annual production of crop residues is around 74 Tg and mainly consisted of straws, roots and stalks that originate from corn (40.6%), wheat (24.2%) and rice (15.74%) (Medina et al., 2015). Fruit and vegetable wastes (FVW) can be defined as inedible parts of plants, that are discarded during their collection (Plazzotta et al., 2017). Between 2000 and 2019 total world production of primary crops increased by 53%, and reached 9.4 billion tones. Data related to the world vegetable production showed its fast growing up to 65% between 2000 and 2019, while fruit production rose, slightly slower, and went up 54%. The main world food products are cereals, which account for almost a third of all crops produced in 2019. After them follow sugar crops (24%), vegetables (12%), oil crops (12%), fruits (9%) and roots and tubers (9%) (FAOSTAT, 2020). Presented data clearly show that increased production of cereals, vegetables and fruits worldwide will lead to an increase of waste production that will reach enormous numbers. Although aforementioned high cereal production leads to a high waste accumulation, these wastes have mostly been utilized in the production of bioethanol and cellulose (Singhania et al., 2014; Thakur et al., 2020; Xu et al., 2020). Hereof, it is crucial to valorize wastes by a detection and quantification of their BACs, and to find the best methods and optimized conditions for their extraction. Several important examples of efficient valorization of agricultural waste with detailed information on extraction process and chemical profile is given in **Table 6**. The costs of underutilized crop waste exploitation could be covered by a development of added value functional products with BACs extracted from wastes and with their potential broad applications.

Table 6. Agricultural waste as source of important BACs

Plant residue	Extraction technique	Processing parameters	Major compounds	Conclusion remarks	References
Sweet potato (*Ipomoea batatas* L.) leaves	*CE* Conventional extraction	-Stirring at 150 rpm -t = 12 h -T = 25± 1°C -S/L (w/v) = 1:20	HPLC-QTOF-MS/MS: -Caffeic acid;	-50% acetone gave the highest solvent efficacy (33.4%, yield of	(Fu et al., 2016)

	-Stirring (Incubator shaker THZ-82A, Jinchengguosheng, Jiangsu, China) -Solvents: water,methanol/water (50,70,90% v/v), ethanol/water (50,70,90% v/v), acetone/water (50,70,90% v/v).	Centrifugation (621.6*g*) -Re-extraction at the same conditions -Evaporation (RE-52A, Yarong, Shanghai, China) at 50 ℃ -Freeze-drying	-Caffeoylquinic acid; -Dicaffeoylquinic acid; -Tricaffeoylquinic acid	extract) followed by 50% ethanol (extract yield 32.4%) -70% ethanol was the most effective for flavonoid (3.4 mg QE/g d.w.) and total anthocyanin (36.5 mg c-3-gE/100g d.w.) content	
	Sample pretreatment by SOX - Soxhlet extraction -Solvent: diethyl ether -Drying at 50 °C in oven *Ethanol extraction* *DHPM (Dynamic high pressure microfluidization)* extraction -MC – ethanol crude extract pretreated by DHPM -EC - ethanol crude extract without	*Ethanol extraction:* -Solvent: 70% (v/v) -S/L (w/v) = 1:40 -t = 12 h -T = 4 °C -homogenization at 30 MPa (valve homogenizer (GYB60-6S, Shanghai Huadong homogenizer Co., China). *DHPM treatment:* -Emulsion homogenization Homogenizer (M-110EH, Microfluidics Company, USA) at 100 MPa twice. -Extraction by refluxing at 75 ◦C for 2 h twice. -Cooling and centrifugation (4000 rpm, 10 min.). *Purification of extracts* LK001 (Shandong LuKang Medicine group Co. Ltd.,	Not determined	-MC extract obtained by pretreatment with DHPM had the highest flavonoid content (58.4 mg/100 mL) -DHPM pretreatment can be used for increasing yield of the flavonoids	(Huang et al., 2013)

	DHPM pretreatment -ECM - ethanol crude extract with posttreatment of DHPM Purified extracts: -MCP – purified MC -ECP - purified EC -ECMP- purified ECMP	China) macroporous absorption resin in a chromatographic column (φ 16 mm×400 mm). -Eluent: ethanol 70% (v/v) -Flow rate of 1.5 mL/min. -Eluents collection at the flow rate of 2 mL/min.			
	UMSE Ultrasonic-Microwave Assisted Extractor CW-2000 (Shanghai Xintuo Analytical Instruments Co., Ltd, China)	*Ultrasonic parameters:* 50W, 2450 Hz *Microwave parameters:* 100W, 2450MHz, wavelength 0.12 m *Extraction repetition:* 1, 2, 3 times *Solvents:* Ethanol solution: 10% (v/v), 30% (v/v), 50% (v/v), 70% (v/v), 90% (v/v), 100% (v/v) T= 35 °C, 45 °C, 55 °C, 65 °C, 75 °C t=25 s, 50 s, 75 s, 100 s, 200 s, 600 s, 999 s Centrifugation at 7000 rpm, 30 min. Volume was adjusted to 500 mL using 30% ethanol.	HPLC (Shimadzu, Japan) Astragalin, Quercetrin, 4,5-Chlorogenic acid, Isoquercitrin, Tiliroside, Quercetin, 3,4,5-chlorogenic acid, Caffeic acid, Kaempferol, Myricetrin, Rhamnetin.	Maximum extraction efficiency of flavonoids was 91.65 ± 3.37% at following conditions: -S/L (w/v) = 1:40 -T=57°C -t= 76 s -Ethanol concentration 72% (v/v) -Extraction for two times Flavonoid purity after purification reached 76.10 ± 3.11 (%, DW).	(Liu et al., 2018)

		Extracts purification: Liquid-liquid extraction Crude extract was vacuum concentrated to half volume and extracted with the same volume of petroleum ether Water phase was extracted with the same volume of ethyl acetate Ethyl acetate phase was vacuum concentrated and evaporated to dryness-			
Red beetroot (*Beta vulgaris L.*) leaves	*CE* Conventional extraction -Stirring on the magnetic stirrer *Solvents*: -Methanol Solid-liquid ratio (w/v) = 1:6 -Water Liquid volume 100 mL	Recovery of obtained extracts: *Methanol extract* -Centrifugation 10000 rpm, 15 min -Filtration *Water extract* -t= 15 min extraction -Filtration	HPLC analysis 25 phenolic compounds were quantified *Methanol extracts major phenolic compounds* -Protocatchoic -E- vanillic *Water extracts major phenolic compounds* - Pyrogallol - E- vanillic 12 different flavonoids were quantified *Methanol extracts major flavonoid* - vitexin *Water extracts major flavonoid* -Rosmarinic	TPC in water extract was higher (7.9 mg GAE/mg fresh weight) was than in methanol extract (2.6 mg GAE/mg fresh weight) No difference in flavonoid content between extracts was observed Antioxidative activity of water extract was higher	(Zein et al., 2015)

	Low pressure methods *SOX* Soxhlet extraction *MAC* Maceration *UAE* Ultrasound-assisted extraction (QR500, Eco-sonic, Brazil, with titanium tip (0.5 cm inside Solution)) Solvents (added separately) Water; Water: Ethanol= 1:1(v/v) Ethanol, Hexane *High pressure method:* *SSF* Supercritical fluid extraction Cosolvent – CO_2	*SOX* - S/L (w/v) = 1:30 -t = 8 h -T = boiling temperature -Assay performed in duplicate -Residual solvent evaporation (rotary evaporator (Mod. 801 and 802, Fitasom, Brasil) *MAC* - S/L (w/v) = 1:5 -t = four days -T = 25 °C -Manually shaken once a day -Removing residual solvent *UAE* - S/L (w/v) = 1:30 -t = 7 min. -T = 25 °C -Sonication power: 500W -Assay performed in duplicate *SSF* -SW:15 g -p=250 bar -T = 25 °C	GC-MS chemical profile of volatile compounds Alcohols, alkanes, aldehydes and ketones were detected. Representative compounds: SFE-10% ethanol for steam - 1-pentanol. Soxhlet-ethanol from stem - 2-Ethyl-1-Hexanol SFE- 7% ethanol + water (90:10) from Leaves - 2-Ethyl-1-Hexanol Ultrasound-ethanol from leaves - 2-Ethyl-1-Hexanol Soxhlet-ethanol from leaves - Heptadecane or Dodecane	Applied extraction technique influenced affected TPC yield, with The highest TPC content (99±4 mg GAE/g for leaves and 98±6 mg GAE/g for steams). Was achieved when SSF cosolvent EtOH:H_2O (90:10) was applied. The technique efficiency ranged: SFE > UAE >SOX > MAC, and SFE > SOX > UAE >MAC for stems Antioxidant activity results variated depending on applied extraction technique, part of the beetroot (leaves or steams used for extraction), solvent and applied test (DPPH, ABTS, FRAP).	(Lasta et al., 2019)
	PLE Pressurized liquid extraction	T=40°C Flow rate: 3 mL/min Sample weight: 5g Solvent evaporation: rotary evaporator	LC-ESI-MS/MS of leaves and stem extracts obtained using PLE (10 MPa and EtOH)	The highest TPC (252±2 mg GAE/g extract) was achieved by using PLE	(Battistella Lasta et al., 2019)

	Self-assembled PLE apparatus Extraction vessel parameters: 138.2 mL, internal diameter of 20 mm and a height of 440 mm. Conditions for evaluation of overall extraction curve (OEC): 10 MPa, 40 °C and 3 mL/min (ethanol 99.8%).	(Fisatom, model 801, Sao Paulo, Brazil) PLE extraction of leaves -solvent: ethanol -7.5; 10; 12.5 MPa PLE extraction of steams -solvent: ethanol -7.5; 10 MPa PLE extraction of leaves -solvents: ethanol: distillated water mixtures of 90:10 and of 70:30 (v/v).	-33 phenolic compound identified in leaves extract -Major compounds: Ferulic acid, Vitexin, Sinapaldehyde -24 phenolic compound identified in leaves extract -Major compounds: Ferulic acid, Methoxyphenylacetic acid, Vanillic acid	extracting of leaves with ethanol as a solvent and pressure of 10 MPa Using PLE extraction at 10 and 7.5 MPa extract that scored better results in FRAP analysis compared to the BHT were obtained-	
Beet (*Beta vulgaris* L. var. conditiva) leaves	*UAE* Ultrasonic Assisted Extraction Vibra-Cell VCX 750, Sonics & Materials Inc., USA) equipped with a flat tip of 13 mm diameter. Frequency, 20 Hz *MAC* Maceration	UAE - S/L (w/v) = 1:10 -t = 0, 1, 2, 4, 6, 8, 10, 12, 14, 20, 30 min. Sonication power: 35, 50, 100, W -Centrifugation at 11200 *g* for 10 min at 5 °C MAC - S/L (w/v) = 1:10 -t = 0, 1, 2, 4, 6, 8, 10, 12, 14, 20, 30 min. T= 20 °C; 20-65°C -Centrifugation at 11200 *g* for 10 min at 5 °C	Not determined	UAE enhanced bioactive compound recovery and compared to the extraction by maceration, shortened (100 W>50W > 35W) The best polyphenol (14.9 mg/g), betaxanthins (949.1 µg/g) and betacyanins (562.2µg/g) yield were achieved under followed conditions: 90W ultrasound power, 1:20 solid-liquid ratio, 16 min extraction time)	(Nutter et al., 2020)

				best	
Beetroot (*Beta vulgaris*) leaves	*SSF* Supercritical fluid extraction (HPE500, Eurotechnica, Germany, extractor volume of 500 mL) Co-solvent - ethanol	- S/L (w/v) = 1:4 - $scCO_2$ flow rate 0.6 kg/h -p=300 bar; 400 bar -T = 35 °C; 40 °C and 50 °C.	Not determined	The highest TPC (3370.8 g GAE/g d.m) was obtained by using the lowest temperature (35 °C) at the highest pressure (400 bar).	(Goyenechea et al., 2020)
Radish (*Raphanus sativus* L) leaves	Fractionization of radish leaves with different polarity solvents: petroleum ether and subcritical water AB-8 macroporous resins column fractionization of the fraction with the highest antioxidative activity	*Petroleum ether extraction*: - S/L (w/v) = 1:15 -Three times *Subcritical water extraction* polytetrafluoroethylene (PTFE) hydrothermal reactor (inner volume 20 mL) -Three times -Centrifugation (5000 rpm/min, 5 min) -Vacuum filtration -Lyophilization -Extract was re-dissolved in distilled water (200 mL) -Fractionization with chloroform (3×200 mL), ethyl acetate (3×200 mL) and water saturated n-butanol (3×200 mL). Extracts were vacuum dried and freeze-dried Obtained fractions: -CLF - chloroform fraction -EAF - ethyl acetate fraction -WBF - water saturated	LC-MS -Caffeoylmalic acid, -P-coumaroylmalic acid, -Feruloylmalic acid, -Kaempferitrin	EAF fraction had the highest TPC (114.12 ± 1.31 mg GAE/g d.w.) and the highest antioxidative activity (IC_{50} for DPPH was 84.19 ± 2.12 µg/L and 83.27 ± 2.13 µg/mL for ABTS). F2 had the highest TPC (477.95 ± 5.31 mg GAE/g dw), DPPH radicals scavenging activity (IC_{50} of 56.44 ± 1.39 µg/Ml) and ABTS radicals scavenging activity (IC_{50} of 49.73 ± 1.74 µg/mL) F2 exhibited the best protection effects on H_2O_2-induced oxidative damaged of MCR-5 cells increasing the cell viability by	(Luo et al., 2018)

		n-butanol fraction -AQF - aqueous fraction *AB-8* *macroporous resins column fractionization* -0%, 20%, 40%, 60%, and 80% ethanol (v/v) for 4 times of column volume, respectively. -- The eluents were collected, lyophilized and named as F0, F1, F2, F3 and F4, respectively.		24.39%	
	MAC Maceration *TD* Thermal digestion *SOX* Soxlet extraction *UAE* Ultrasound Assisted Extraction *Solvents*: -Ethanol, Methanol, Water, Chloroform *Drying procedure* Rrotary evaporator (Heidolph, R-200, Germany) Freeze dryer (Mitsubishi, Vaco, 5-11, Germany) at -80 ºC.	*MAC:* S/L (w/v) = 1:20 -Shaking, t = 24h -Room temperature -Filtration Evaporation at reduced pressure *TD:* Solid-liquid ratio (w/v) = 1:20 t= 1h, 6h, 24h. T=34-40 °C. Filtration, vacuum evaporation *SOX:* S/L (w/v) = 1:40 t= 24h. Filtration, vacuum evaporation *UAE:* Ultrasonic homogeneisator (Hielscher UP400S) S/L (w/v) = 1:20 t= 1min, 5 min, 10 min. Room temperature, Frequency of 50/60 kHz 20%, 50% and 100% of ultrasound intensity	HPLC liquid chromatography -Quercetin content determination	Ultrasonic Assisted Extraction (UAE) with 50% of ultrasound intensity (Frequency of 50/60 kHz) for 10 min in methanol was the most efficient technique for quercetin extraction (11.8% yield).	(Sharifi et al., 2017)

		Extraction with best solvent (methanol), was performed in 5, 10 and 15 min with different ultrasonic apparatus intensities.			
	SSF Supercritical fluid extraction (HPE500, Eurotechnica, Germany, extractor volume of 500 mL)	- S/L (w/v) = 1:4 - $scCO_2$ flow rate 0.6 kg/h -p=300 bar; 400 bar -T = 35 °C; 40 °C and 50 °C.	Not determined	Increased pressure and decreased temperature (35 ºC/400 bar and 40 ºC/400 bar), gave the highest TPC yield (1375 mg GAE/ 100 g d.w. and 1455 mg GAE/ 100 g d.w. respectively). DPPH radical scavenging activities correlated the TPC content, (359 mg TE/100 g d.m. (35 °C/400 bar) and 403 mg TE/ 100 g d.m. (40 ºC/400 bar)).	(Goyenechea et al., 2018)
Apple (Szampoin, Ozark Gold, Idared cvs.); Pear (Faworytka, Alfa cvs.); Quince (ZM4, CTM6, Vranja cvs.); Apricot (Early orange, Wczesna z Modern, Harcot cvs.); Peach (Harbringer, Kijowska wczesna, Candor cvs.); Plum (Węgierka zwykła, Amers, Opal cvs.); Sweet cherry (Rivan, Techlovan, Summit	*CE* Conventional extraction -Shaking (DOS-10L Digital Orbital Shaker, ELMI; Riga, Latvia) -Solvents: hexane:acetone:methanol (2:1:1(v/v/v))	-S/L (w/v) = 1:30 -Shaking at 300 rpm -t=30 min -No light -In the presence of 10% $MgCO_3$ and 1% BHT -Centrifugation (MPW-55; Warsaw, Poland) -Residue re-extraction 4 times -Evaporation to dryness under nitrogen (XcelVap®, Thermo Scientific)	LC-PDA-qTof-ESI-MS (identification) and UPLC-PDA (quantification) Identified: -7 carotenoids belonging to caroten and xantophylls -16 chlorophylls	-The best sources of chlorophylls are apple, sour cherry and apricot leaves (mean 404.8, 388.7 and 364.5 mg/100 g dw, respectively). -The best sources of carotenoids are sweet and sour cherry leaves as (831.4 and 1162.0 mg/100 g dw,	(Wojdyło et al., 2021)

cvs.) and Sour cherry (Łutowka, ´ Turgieniewka, Topaz cvs.) leaves				respectively).	
Apple (Ozark Gold cv.) leaves	UAE Ultrasonic-assisted extraction -Solvent: 80% aqueos methanol and 1% of HCl	-S/L (w/v) = 1:20 -Stirring: slight -Sonication t=30 min -Left in the dark for 24 h and 4°C -Sonication -Centrifugation 10 min, 20878xg	Identification of Polyphenols: LC–MS and HPLC, Polymeric Procyanidins Analysis: HPLC-FL -Detected compounds: five flavan-3-ol; two dihydrochalcones; three hydroxycinnamates ; four flavonols. -dihydrochalcone phloretin-20 -O-glucoside predominted (11% of d.w.)	Leaves had much higher content of polyphenolic compounds and antioxidant capacity than fruits.	(Wojdyło and Oszmiański, 2020)
Apricot (*Prunus armeniaca* L.) leaves	UAE Ultrasound-assisted extraction - Solvents: methanol, n-propanol, isopropanol (40, 70 and 99% (v/v)) Ethanol (40, 70, 96% (v/v)).	S/L (w/v)=1:20 -Sonication: Frequency: 40kHz t=15, 30, 60 min	Not determined	-Methanol and ethanol solvents gave the best DPPH radical scavenging activity (>70%) -15 min of sonication in concentrated methanol reached 84.66% of the activity. -60 min of sonication in 40% (v/v) ethanol had 84.55% of the activity.	(Pietrzyk and Klimowicz, 2021)
Peach (*Prunus persica* (L.) Batsch)leaves	CE Conventional extraction - Solvent: acetone:methanol:water:formic acid (40:20:20:0.1)	CE -S/L (w/v) = 1:6 - Centrifugation (5000 rpm, 15 min, 4 °C) -Residue re-extraction three times, same solvent	Not determined	-SSF extract showed better NO inhibitory activity (IC_{50}=9.3 µg/ml) and better cytotoxic activity against HeLa, MPanc-96 and MCF-7 (IC_{50}	(Koyu et al., 2019)

	SSF Supercritical carbon dioxide (CO2) extraction (Thar Designs SFE-100, U.K.)	SSF -t = 60 °C, -p=150 bar, -co-solvent 6% ethanol -Dynamic extraction (t= 60 min, flow rate of 15 g/min)		values of 12.22 , 28.17 and 35.51 µg/ml respectively), than CE extract (IC_{50}≥50 µg/ml) -MIC values for SSF were lower for *E. coli* and *S. aureus* than for CE extract. -TPC for SSF and CE were 82.56 and 106.02 GAE/g extract, respectively	
Grapes (*Vitis vinifera* L.; varieties-white: Maraština, Pošip; red: Lasin, Merlot, Syrah, Vranac) leaves	CE Conventional extraction -Solvent: ethanol/water 80/20 (v/v))	-S/L (w/v) = 1:5 -t=60 min -T=60 °C -Filtration -Washing 2x with 25mL of solvent -Drying under vacuum, 50 °C -Extract was redissolved in methanol-water (50:50 (v/v))	HPLC-RP-PDA -Flavan-3-ols: (+)-catechin and (−)-epicatechin -Flavonols: apigenin, kaempherol, quercetin, myricetin, quercetin-4-glucoside, and rutin -Stilbenes: cis- and transresveratrol monomers and astringin	-TPC ranged from 9.4 to 23.4 g GAE/kg of dry leaves - September LEs, were potently active and exhibited strong and concentration-dependent DPPH radical-scavenging ability. - increase in FRAP was noticed especially in September (141 ± 33 mmol TE/L	(Katalinic et al., 2012)

[a] CE, conventional extraction; DHPM, Dynamic high pressure microfluidization; HPLC-QTOF-MS/MS, High-performance liquid chromatography coupled to diode array detector, electrospray ionization quadruple time-of-flight tandem mass spectrometry; MAC, Maceration; SOX, Soxlet extraction; SW, Sample weight; TD, Thermal digestion; UAE, Ultrasound Assisted Extraction; UMSE, Ultrasonic–microwave synergistic extraction; HeLa, Human cervical cancer cells; MPanc-96, Human pancreatic cancer cells; MCF-7, , human breast adenocarcinoma; MIC, Minimum inhibitory concentration; S/L= solid/liquid ratio; t, Time; T, Temperature, ABTS, 2,2'-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid; DPPH, 2,2-Diphenyl-1-picrylhydrazyl; BHT, Butylated hydroxytoluene.

The world production of beetroot in 2017 reached 301 million tons, with Europe that ranks first (69% of the world production, *i.e.,* 207.9 million tons) followed by Asia (42.7 million tons), America (34.3

million tons) and Africa (15.9 million tons) (FAOSTAT, 2019). About 50% of the whole plant represents beet aerial parts, *i.e.,* leaves and steams, which are usually used to feed animals, as organic fertilizer or are discarded as waste, and thus, one half of the produced biomass is lost after harvest (Biondo et al., 2014; Nutter et al., 2020). Main producer of red beetroot (*Beta vulgaris L.*) that is beet (*Beta vulgaris* L. subsp. *vulgaris*) subspecies is Brazil, which covers 77% of world production. Beetroot aerial parts have high content of minerals, vitamins fatty acids, especially omega-3 polyunsaturated fatty acids whose consumption can prevent development of rheumatoid arthritis, coronary diseases, colon, prostate, and brain cancer (Biondo *et al.*, 2014). Using conventional stirring extraction of beetroot leaves Zein, Hashish and Ismaiel (2015) showed that water extract had higher TPC (7.9 mg GAE/mg f.w.b.) compared to methanol extract (2.6 mg GAE/mg f.w.b.). Regarding flavonoids, no difference between samples was observed (0.44 mg/g f.w.b. in water extract and 0.43 mg/g f.w.b in methanolic). Consequently, DPPH radical scavenging activity of water beetroot extract was higher (86.6%), compared to methanol extract (82.4%). HPLC analysis quantified 25 phenolic compounds in both extracts, and 12 different flavonoids, and established differences in the content of detected compounds between samples. This is most probably due to the differences in the polarity of applied solvents and the nature of phenolic compounds present in leaves. In addition, cytotoxic activity of beetroot leaves against breast and liver cancers was reported. Total acceptability of an orange or mango juices fortified with beetroot leaves have significantly differed compared to pure juices. Nevertheless, these juices did not have noticeable impact on sensory properties (Zein et al., 2015).

Lasta et al. (2019) compared low pressure methods (Soxhlet extraction (SOX), maceration (MAC) and ultrasound-assisted extraction (UAE)) and high-pressure method - supercritical fluid extraction (SFE) for extraction of beetroot leaves and stems. For low pressure methods, water; water/ethanol mixture 1:1 (v/v); ethanol and hexane were used as solvents, while for SFE, CO_2 and cosolvents (ethanol, and ethanol/water mixture (90:10 (v/v)) were applied. TPC content varied depending on the applied method from 1.92 to 99 mg GAE/g extract for leaves, and 4.26 to 98 mg GAE/g extract for stems, pointing to SFE with cosolvent ethanol/water (90:10) as the best method. The efficiency of applied extraction techniques on TPC recovery from leaves were ranged as follows: SFE > UAE > SOX > MAC, and SFE > SOX > UAE > MAC for stems. Application of EtOH as a solvent in SOX, MAC and UAE, gave better results than SFE for DPPH radical scavenging activity. On the other hand, SFE provided high DPPH activity when ethanol and ethanol/water mixtures as cosolvents were used (EC_{50} from 150 ± 7 to 346 ± 6 μg/mL). This is, most probably, due to a selective extraction of low molecular weight phenolics that contributed to the higher antioxidative potential, than carbohydrates fraction which was extracted by other methods. Results for FRAP analysis of leaves extracts were the best when MAC with ethanol (259 ± 5 μ MTEAC/g extract) and SFE with 10% ethanol (240 ± 3 μ MTEAC/g extract) were applied. The same procedure for SFE gave the best results for stems (633 ± 33 μ MTEAC/g extract). The best ABTS results were obtained using SOX extraction with ethanol (586 ± 82 μ MTEAC/g extract for beetroot leaves, and 730 ± 14 μ MTEAC/g extract for stems), differing statistically from other results.

Battistella Lasta et al. (2019) used pressurized liquid extraction (PLE) with different process parameters regarding applied pressure (7.5, 10 and 12.5 MPa) and solvent (ethanol, ethanol: distillated water mixtures of 90:10 and 70:30 (v/v), to extract beetroot aerial parts. They showed that ethanol is more selective solvent, giving the higher TPC content (252 ± 2 mg GAE/g) than ethanol/water = 70:30 mixture (7 ± 1 mg GAE/g). TPC for stems obtained using PLE with ethanol was

lower compared to leaves, (up to 16 ± 1 mg GAE/g extract), while significant difference in TPC content regarding applied pressure for their extraction was not established. Liquid chromatography, identified 33 phenolic compounds in leaves extract, and 24 in stem extract. The most abundant compounds were ferulic acid, vitexin, and sinapaldehyde that have potential application in nutraceutical, functional food and herbal production. Good DPPH radical scavenging activity was observed, with EC_{50} similar to BHT by using PLE at 7.5 and 10 MPa and pure ethanol or ethanol/water mixture. The highest values for ABTS antioxidant method were achieved when 12.5 MPa for leaves (823 µmol Trolox/g extract) and 10 MPa for stems (49.7 µmol Trolox/g extract) were applied. Obtained result for 12.5 MPa and ethanol as solvent was higher than in the research by Lasta et al. (2019) when the same material was extracted by SOX and UAE techniques. Additionally, results for ABTS were better in comparison to BHT, widely applied synthetic antioxidant (Battistella Lasta et al., 2019). The results of FRAP analysis of the samples obtained using PLE extraction of leaves showed that applying the ethanol at 10 and 7.5 MPa gave better results compared to the BHT as a control. Also, higher FRAP values were obtained using PLE extraction at the mentioned conditions compared to values reported by Lasta *et al.* (2019) for SOX and UAE techniques.

Nutter et al. (2020) used UAE of beet leaves (*Beta vulgaris* L. var. conditiva) for the recovery of polyphenols and betalains and compared it to the extraction by maceration (MAC). Water was used as solvent and different process parameters were tested. It was revealed that UAE enhanced bioactive compounds recovery and shortened extraction time for 8, 6 and 2 min with ultrasound treatments of 35, 50 and 100 W, respectively. The yields were comparable to 30 min of maceration at 20 °C. UAE was optimized, using Box-Behnken design and Response Surface Methodology, and the optimal conditions were found to be: 90 W ultrasound power, 1:20 solid-liquid ratio and 16 min of the extraction time, to obtain the best polyphenol (14.9 mg/g), betaxanthins (949.1 µg/g) and betacyanins (562.2 µg/g) yields.

Goyeneche et al. (2020) also used SFE with ethanol as a cosolvent, and different extraction parameters (pressures: 300 and 400 bar; temperatures: 35, 40 and 50 °C) to extract phenolic compounds. The best results for TPC (3370.8 g GAE/g d.m.) were obtained by using the lowest temperature (35 °C) and the highest pressure (400 bar), achieving at the same time, the highest value of antioxidative capacity assayed by DPPH test (1454.0 g TE/g d.m.).

Sweet potato (*Ipomoea batatas* L.) ranks fifth as the most essential food crop in developing countries after rice, wheat, maize and cassava (CIP, 2019, p. 4). Its annual world production amounts more than 90 million tones. (FAOSTAT, 2020). The sweet potato roots are consumed, while their leaves are mostly (95-98%) discarded, causing huge waste (Alam, 2021). Leaves of the sweet potato contain significant amounts of nutrients and bioactive compounds (i.e., polyphenols, proteins, dietary fibers, carotenoids, vitamins, minerals), and thus can be considered as underutilized agricultural waste that can be used for the creation of value-added products (Fu et al., 2016; Huang et al., 2013). Among different solvents, (water, aqueous methanol, aqueous ethanol and aqueous acetone) Fu et al. (2016) revealed that the use of 70% ethanol gave the highest total flavonoid (3.4 mg QE/g d.w.) and total anthocyanin content (36.5 mg c-3-gE/100 g d.w.) in obtained extracts. On the other hand, 50% acetone was the most effective for yield (33.4% in d.w.) and TPC (43.8 mg CAE/g d.w.) recovery. Additionally, 50% acetone extract exhibited the highest DPPH radical scavenging activity (36.6 mg VcE/g DM), reducing power (OD_{700} = 0.81 at 1.2 mg d.w./mL) and HO scavenging activity (27.8 mg VcE/g d.w.). HPLC analysis identified 14 phenolics in 50% acetone extract where caffeoylquinic acid

(CQA) and quercetin derivatives were the most dominant. Huang et al. (2013) studied the impact of dynamic high pressure microfluidization (DHPM) assisted extraction, on the isolation of flavonoids from sweet potato leaves. Extract obtained using pretreatment with DHPM had the highest flavonoid content (58.4 mg/100 mL), suggesting that its application can be used to increase flavonoid extraction. Additionally, extracts prepared using DHPM-assisted extraction exhibited higher DPPH radical scavenging activity compared to ones obtained without pretreatment or using DHPM as posttreatment. To extract flavonoids from sweet potato leaves Liu et al. (2019) optimized ultrasonic–microwave synergistic extraction (UMSE) by response surface methodology. The optimal conditions to obtain extraction efficiency of even 91.65% were: solid-liquid ratio (w/v) 1:40, temperature 57 °C, extraction time of 76 s, and 72% (v/v) ethanol, two times. After extract purification, the purity of flavonoids reached 76.10 ± 3.11%, d.w., and HPLC analysis quantified 11 different compounds. The Fe^{3+} scavenging capacities of flavonoids reached maximum of 76.22 ± 0.49% at 20 µg/mL, and their activity was better than the activity of soy isoflavones, *Ginkgo biloba*, and propolis flavone.

Radish (*Raphanus sativus* L.) is a root vegetable that consists of edible enlarged hypocotyl and leaves that are not consumed and are usually trimmed, representing a waste that can be used as a source of bioactive compounds (Tsouvaltzis and Brecht, 2014). Fractionization of radish leaves with different polarity solvents was the focus of the research conducted by Luo et al. (2018). Four fractions were obtained: chloroform (CLF), ethyl acetate (EAF), water saturated n-butanol (WBF) and aqueous (AQF) fractions. Among them, the EAF had the highest TP (114.12 ± 1.31 mg GAE/g d.w.) and antioxidative activity (IC_{50} values 84.19 ± 2.12 µg/L for DPPH and 83.27 ± 2.13 µg/mL for ABTS). The same extract showed the highest protective activity on the human fetal lung fibroblast (MRC-5) damaged cells, increasing their viability by 19.06%. By using AB-8 macroporous resins column and 40% (v/v) ethanol, EAF sub-fraction F2 was obtained, with the highest TPC (477.95±5.31 mg GAE/g d.w.), DPPH radical scavenging activity (IC_{50} of 56.44±1.39 µg/mL) and ABTS radical scavenging activity (IC_{50} of 49.73±1.74 µg/mL). F2 fraction also exhibited protective effect on the H_2O_2-induced oxidative damage of MCR-5 cells. As the major phenolic compounds caffeoylmalic acid, p-coumaroylmalic acid, feruloylmalic acid and kaempferitrin were quantified by HPLC analysis.

Comparison on efficiency of MAC, TD, SOX and UAE and solvents (ethanol, methanol, water and chloroform) to extract quercetin, a flavonoid from radish leaves, was performed by Sharifi, Mahernia and Amanlou (2017). The UAE method at 50% of ultrasound intensity (frequency of 50/60 kHz) applied for 10 min in methanol was the most efficient quercetin extraction technique, achieving yield of 11.8%. Regardless of the applied technique, methanol was the most efficient solvent for quercetin extraction.

Goyeneche et al. (2018) used supercritical CO_2 (0.6 kg/h flow rate) and ethanol as a cosolvent as alternative to conventional methods to extract bioactive compounds from radish leaves. Extraction curves were obtained at different pressures (300 and 400 bar) and temperatures (35 °C, 40 °C and 50 °C). The best results were obtained at increased pressure and decreased temperature (35 °C/400 bar and 40 °C/400 bar), when contents of TP were 1375 mg GAE/ 100 g d.w. and 1455 mg GAE/ 100 g d.w., respectively. DPPH radical scavenging activity was in correlation with the TPC content, with obtained values 359 mg TE/100 g d.m. (35 °C/400 bar) and 403 mg TE/ 100 g d.m. (40 °C/400 bar). Additionally, both extracts did not show cytotoxicity on Bone Marrow-derived Dendritic Cells (BMDCs) up to 200 µL after 18 h of incubation. Also, maturation of BMDCs was not promoted, and

thus, obtained extracts are the promising source of antioxidative and anti-inflammatory agents that can be used in the pharmaceutical and food industries.

Fruit tree leaves are non-conventional parts of plants that are important sources of phenolic compounds and isoprenoids (e.g., chlorophylls and carotenoids). The content of these components in fruits and their parts depend on various factors including maturity, genotypes and cultivars (Pietrzyk and Klimowicz, 2021; Wojdyło et al., 2021). Polyphenols' content, composition and the way of extraction influence antioxidant potential of fruits and their wastes (Pietrzyk and Klimowicz, 2021). Wojdyło et al. (2021) collected different fruit tree leaves (FTL) from apples, pears, quince, peach, apricot, plums, sour and sweet cherries in spring and autumn to extract, identify and quantify chlorophylls and carotenoids. Extraction was performed using conventional stirring by hexan:acetone:methanol (2:1:1, v/v/v) mixture. The best source of chlorophylls was FTL of apple, sour cherry and apricot (mean 404.8, 388.7 and 364.5 mg/100 g d.w., respectively), while as the best source of carotenoids determined was sweet and sour cherries (831.4 and 1162.0 mg/100 g d.w, respectively). It was found that FTL are good sources of both components, especially in spring with contents between 5 and 20% higher than that in autumn leaves. FTL have good potential to be applied in cosmetics, pharmaceutical and food industries.

To extract polyphenols, Wojdyło and Oszmiański (2020) used apple (*Malus domestica*) leaves samples collected during different stage of fruit development, and extracted them by stirring (80% aqueous methanol solution, with 1% of HCl) followed by 15 min of sonication. After 24 h at 4 °C samples were sonicated again, and supernatant was recovered by 10 min centrifugation (20.878 g). Qualitative LC–MS analysis detected the presence of 17 phenolic compounds, while HPLC-MS quantification revealed that the most abundant component in apple leaves was dihydrochalcone, phloretin-2'-O-glucoside (over 11% of dry weight), known to be a pharmaceutical component used in hypoglycemia treatment. It was found that apple leaves were better source of TPC and quercetin glycosides than apple fruits. In addition, leaves extracts had better antioxidant capacity measured using DPPH radical scavenging and ABTS assays, than apple extracts and lower variations in capacity between samples collected in different stages of development (Wojdyło and Oszmiański, 2020). Pietrzyk and Klimowicz (2021) evaluated the effect of applied extraction condition of apricot leaves on the antioxidant activities of the obtained extracts, assayed by DPPH method. UAE (40 kHz, applied for 15, 30 or 60 min) coupled with methanol, ethanol or *n*-propanol of different strengths. The highest DPPH radical scavenging activities were achieved with concentrated methyl alcohol and 15 min of sonication (84.66%) and 40% (v/v) of ethanol with 60 min of sonication (84.55%) (Pietrzyk and Klimowicz, 2021).

Peach (*Prunus* species) cultivation requires intensive pruning and significant amounts of leaves are left as waste. Peach leaves are the source of phenolic compounds such as: caffeic acid, chlorogenic acid, coumaric acid, kaempferol etc. (Koyu et al., 2019). SFE optimization study was done by Koyu et al. (2019) by Box-Behnken design, tested independent variables were co-solvent ratio (6-20%), temperature (40-80 °C) and pressure (150-300 bar) for the highest TPC and radical scavenging activity. The best parameters for the extraction were 60 °C, 150 bar and 6% ethanol as co-solvent with the duration of 60 min and flow rate of 15 g/min. Remarkable potency of such prepared extract was against HeLa (human cervical cancer cells), MCF-7 (human breast adenocarcinoma) and MPanc-96 (human pancreatic cancer cells) and for NO inhibitory activity, while in comparison with conventional (acetone:methanol:water:formic acid, 40:40:20:0.1) extract these activities were

weaker. Both extracts (SFE and conventional) showed similar antimicrobial activity, while SFE extract was more potent against *E. coli* and *S. aureus*.

The study of Katalinic et al. (2012) investigated biological activities of polyphenolic extracts from six *Vitis vinifera* L. varieties leaves (LEs) picked in three different cultivation phases. Extraction was performed using ethanol solvent (ethanol/water: 80/20 v/v) at 60 °C for 60 min. Extracts were recovered by filtration, washed two times with ethanol solvent and dried under vacuum at 50 °C. The obtained results showed that the TPC (up to 23.4 g GAE/kg of dry leaves) was dependent on variety and time of picking since TPC and flavonoid content in September were 40.5 g GAE/L, and 15.5 g GAE/L, respectively, which was about 4-foulds higher than in May. The principal flavonoid component in all LEs was (+)-catechin, and the dominant flavonol was quercetin (up to 35 mg/kg dry leaves). Antioxidant activity was a result of an additive or synergistic activity of all phenolic compounds, while antimicrobial activity (MIC between 0.38 and 2.24 mg GAE/mL) was better in extracts obtained in August and September when higher content of flavonols and stilbenes was found. The leaves picked in September, after grape harvest, are recommended, from economic point of view, for the production of extracts with antioxidant and antimicrobial activity and the application to prevent food deterioration and/or microbial spoilage.

Considering previously analyzed scientific papers, it is obvious that agricultural waste extracts represent significant source of underutilized bioactive components (*i.e.* phenolic compounds, carothenoids, chlorophills) with proved bioactivity (antioxidant, antimicrobial, anticancerogenic). The application of those extracts in pharmaceutical or food products is not sufficiently examined and future research should be undertaken in that direction.

7. Challenges and opportunities

Scientific community and food sector specialists face tremendous challenge in terms of ensuring the food safety and security, as well as introducing Industry 4.0 tools to reduce losses and waste of food. Furthermore, novel solutions should provide acceptable and economically competitive products and functional foods fortified with bioactive compounds and antioxidants that promote health and support consumers' immune system (Galanakis, 2020). Post-pandemic era brings both challenges and opportunities for the development of new innovative nutraceuticals that may lead to techno-socioeconomic disruption in the food ecosystem throughout the supply chain and marketplace (Galanakis et al., 2020). Valorization of a vast range of bioresources towards the production of BACs has been highlighted as one of the main Industry 4.0 applications and innovations together with lab-grown meat and plant-based alternatives of meat (Galanakis et al., 2021). The development of technologies that will enable the efficient use of generated bio-waste and side streams is on the rise, and research is increasingly turning to "green" and environmentally friendly techniques. The valorization concept proposed in Figure 1 adheres to the requirements of the circular economy and offers solutions for a smart industry that saves natural resources and generates a minimum of waste, by applying "green" extraction technologies. It shows the planned route of valorization of the agri-food waste, selected by-products of the food industry, as well as unused plant material that is often found in Serbia (such as plant dust of medicinal plants or wild growth plants), in order to obtain valuable BACs that can be further incorporated into myriad products of various industries, especially pharmaceutical, cosmetic and food industries. Modern extraction techniques that rely on the use of green solvents are particularly suitable in order to fulfill the tendency of consumption towards

health-safe products. In this regard, the planned exploitation of the reviewed plant materials should be explored towards utilization of emerging extraction techniques and with the use of new generation solvents, primarily natural deep eutectic solvents (NADES), subcritical water and supercritical carbon dioxide. The intended solvents have the green character and thus they are subject to all environmental standards and laws, at the same time providing high yields and high selectivity of the process. All of these solvents offer the possibility of modulating their solvating properties by changing the experimental conditions, allowing fine-tuning of the process selectivity. In addition, the extracts obtained with these absolutely non-toxic and environmentally friendly solvents can be directly incorporated into the final products without prior purification, thus ensuring the economy of the process. Emphasis should be also put on separation and purification of crude extracts. According to Galanakis (Galanakis, 2015), ultrafiltration, nanofiltration and other membrane technologies are among the key physicochemical and non-destructive techniques that could be applied for separation of macro- and micro-molecules towards further purification in downstream processing. The obtained extracts of lipophilic and hydrophilic components should be further characterized in detail in terms of chemical and biological characteristics, prior to their inclusion in food, cosmetics or pharmaceutical formulations.

In certain cases extracts or by-products which are used as raw material for extraction of BACs must be subjected to safety examination which increases the cost of production and delays placement on market. For example orange peels are by-product rich in terpenoids, pectins and polyphenols which could be successfully extracted using green extraction techniques. However, orange peels may contain significant amount of pesticides (Li et al., 2012), which were used to protect plants from insects and diseases during vegetative and post-harvest period. Furthermore, in order to reduce potential for microbial spoilage it is sometimes required to reduce water content in by-products/extracts. By doing so, by-products such as coffee cascara, which are rich in nutirents (sugars and proteins) may undergone Malliard reaction when dried and transform into melanoidins and finally to potential carcinogen acrylamide. A recent study done by Iriondo-DeHond et al. (Iriondo-DeHond et al., 2020) demonstrated that levels of acrylamide in new coffee cascara-based beverage was lower then limits established by the European Union (EU) for coffee and instant coffee (450 and 900 μg/kg), however this needed to be tested prior to any further use. This indicated that extracts shouldn't only be tested for safety issues but also aligned with different regulations. For example in the EU, if obtained extract is further used in food production its usage needs to be in alignment with the European Commission (EC) Regulation 178/2002. On the other hand, if those plant extracts were not used in food industry prior to May 1997, they are considered as "new ingredients" and their safety issues needs to be tested prior to commercialization on the EU market. In the EU, there are also other regulations/directive which are applied to nutraceuticals (Directive 2002/46/EC), food by-products (EC Regulation 315/93/EEC), food by-products as food additives (EC Regulation 1333/2008). Recovered extracts needs also to be aligned with maximal levels for contaminants in food products (EC Regulation 1881/2006) (Socas-Rodríguez et al., 2021). Further emphasis should be put on several limitations which could be associated with the processing of by-products and waste streams. By-products generated within the food industry have to be further processed prior to the further extraction steps. Drying is often demanded in order to preserve raw material from spoilage and adding this step in the production will certainly increase the cost of processing. Having that in mind, herbal dust from filter tea might be an interesting choice for extraction since additional processing steps with this by-product are not necessary (B. Pavlić et al., 2016b), thus it could be directly

extracted by selected extraction technique. It has been highlighted that novel extraction techniuques could be followed by different drawbacks regarding food safety, efficiency, energy, and operational cost and special emphasis should be put on the disruptive effects on BACs which are occasionally associated with ultrasounds and cold plasma (Galanakis, 2021). Furthermore, majority of valorization examples given in this review are performed at a laboratory level using novel extraction techniques. This raises further limitations due to limited availability of emerging techniuques at industrial scale and processing cost which should be further evaluated. This suggests that research obtained at a laboratory level should be followed by the techno-economic study which includes scale up on the industrial scale. One good example is research by Prado et al. (Prado et al., 2012) where scale up and economic analysis were evaluated for the supercritical fluid extraction of grape seed and the production of grape seed oil. Addition of extracts obtained from by-products or waste in food, nutraceuticals or cosmetic products might impact consumer's preferences and perception. Consumers might be suspicious and hesitant to use these products, therefore, special emphasis should be put on introducing the safety studies to the proposed valorization concept. Extracts could also provide significant impact on sensory properties of food products which could also cause unacceptance of products by the consumers. This suggests that studies focused on the addition of these extracts in food or cosmetics should include sensory evaluation. Several studies suggest that extracts with strong flavor and aroma could still be included in food products in concentration high enough to prevent oxidation and microbial spoilage and at the same time sufficiently low so that the impact on sensory properties of food products are minimal (Šojić et al., 2018).

8. Conclusions

The agri-food industry generates a vast amount of by-products that is considered as an important problem worldwide due to its harmful environmental impacts. Furthermore, food industry by-products and agricultural waste are usually under valorized although several studies have suggested that it could be a remarkable source of a wide range of BACs which have been extensively studied for their health benefits and important biological activity. Reviewed studies highlighted that valorization of agri-food by-products and side streams provides solutions for reduction of waste streams through recovery of value-added products from sustainable resources such as under-utilized plant resources, food industry by-products and agricultural waste. Furthermore, recent studies suggested that novel extraction techniques could address several major challenges associated with conventional techniques and procedures. Strong scientific evidence supporting the efficiency of emerging extraction techniques for production of high-quality extracts with improved bioactivity suggests that different applications need to be explored urgently and special emphasis should be placed on utilization of supercritical fluids and NADES as extraction solvents in order to enhance extraction rates, improve stability of BACs and at the same time preserve "green" nature of the process. This will allow sustainable production of value-added products, designed for high-value applications in pharmaceutical and/or cosmetics and/or food sector.

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