Subcritical-Water Extraction of Natural Products

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Contributor: Yu Yang

Subcritical water refers to high-temperature and high-pressure water, but below water's critical point of 374 °C and 218 atm. A unique and useful characteristic of subcritical water is that its polarity can be dramatically decreased with increasing temperature. Therefore, subcritical water can behave similarly to methanol or ethanol. This makes subcritical water a green extraction fluid used for a variety of organic species.

Keywords: natural products ; subcritical water extraction ; alkaloids ; glycosides ; flavonoids ; essential oils ; quinones ; terpenes ; lignans ; organic acids ; polyphenolics ; steroids ; carbohydrates

1. Introduction

Among the various new green extraction and separation technologies developed recently, subcritical water extraction (SBWE) is the most promising one. Subcritical water refers to the liquid water at temperature and pressure below its critical point (Tc = The pressure of the subcritical water must be higher than the vapor pressure at a given temperature to keep water in the liquid state. With the increase of temperature, the physical-chemical properties of subcritical water change drastically.

2. Compounds Extracted by Subcritical Water

2.1. Flavonoids

Flavonoids, also known as bioflavonoids, are widely found in plants and berries. They are important natural compounds in human diets. They have been used to prevent and treat cardiovascular diseases. In addition, they have strong antioxidant activities and antibacterial activities. When high-flavonoid apples were fed to healthy mice, decreases in some inflammation markers were reported [1].

Generally speaking, flavonoids belong to phenols. Since they are widely investigated, they can be presented separately from phenols. Flavonoids have the general structure of a 15-carbon skeleton by connecting two benzene rings with a heterocyclic ring. The basic nucleus is 2-phenylchromone. Flavonoid compounds are usually poorly soluble in ambient water and most organic solvents. **Table 1** summarizes SBWE of flavonoids from plant materials.

Table 1. SBWE of flavonoids.

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Panax ginseng C.A. Meyer	stem leave	TP and flavonoids	antibacterial	110 and 165 °C, 15 min 190 °C, 10 min	TEM, UV	heating (water 95.4%; ethanol 91.3%)	[2]
Chamomilla matricaria L.	flowers	TP, TF, 18 polyphenolic compounds, apigenin	antioxidant, enzyme inhibitory activity	65–210 °C, 5–60 min 1:30–1:100 g/mL	TLC, UV, HPLC-MS		[3]

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Allium cepa	onion wastes	quercetin-4′-glycoside, quercetin, etc.		40–160 °C, 5 min, 5 MPa, 1–10 mm, pH 3.0–7.0	LC-MS/MS HPLC-UV	convention (methanol and hydrochloric acid 94.3%)	[4]
Crocus sativus L.	stigmas	TP, dodecane, γ-terpinene, tetradecane, etc.	antioxidant (DPPH, FRAP), antibacterial	100–180 °C, 10–30 min, 1:10 g/mL	GC/MS, UV-vis		[5]
Saururus chinensis, etc.	skin, leave, peel, etc.	quercetin, isorhamnetin, kaempferol, isoquercitrin, etc.		10 MPa, 110–200 °C, 5–15 min	HPLC		[6]
Camellia sinensis	leaves	epigallocatechin gallate		80–120 °C, 3–7 min, 40–60 mL/g	HPLC	convention (water 87.6%)	[7]
Origanum vulgare L.	leaves	TP, flavanone, flavone, flavanol	antioxidant (DPPH, TEAC, ABTS)	10.34 MPa, 30 or 15 min 25–200 °C	HPLC-DAD, UV		[8]
orange	peels	reducing sugar, TP, pectin, hesperidin, narirutin	antioxidant (DPPH, FRAP)	110–150 °C, 10–30 mL/min 10 MPa	HPLC, UV-vis	Soxhlet (ethanol 79.2%), shaker (ethanol 250%), UAE (ethanol 114%)	[9]
orange	peels	flavones, 7-hydroxyflavone		100–150 °C, 0.5 mL/min	GC-FID	UAE (methylene chloride)	[10]
Citrus unshiu Markovich	peels	rutin, naringin, hesperidin, naringenin		0.5–14 MPa, 5–15 min, 100–190 °C	HPLC		[11]
Allium cepa L.	peels	TP, TF, quercetin	antioxidant (DPPH, TBA, FTC)	110 and 165 °C, 15 min, p < 3.4 MPa	HPLC, UV	heating (ethanol 153%; water 45.6%)	[12]
Hippophae rhamnoides	leaves	TP, TF, isorhamnetin, kaempferol, quercetin	antioxidant, cytotoxicity	25–200 °C, 15 min, 10.34 MPa	HPLC, UV, FM	maceration (water 21.3%), Soxhlet (ethanol 64.6%)	[13]
Allium cepa L.	peels	TP, TF, kaempferol, quercetin	antioxidant (DPPH)	170–230 °C, 3 MPa, 30 min, pH 2–10	HPLC, UV-vis	heating (ethanol 26.7%)	[14]
Achillea millefolium L.	herbal dust	TP, TF, HMF, chlorogenic acid	antioxidant (DPPH, TEAC, ABTS)	120–200 °C, 10–30 min 0–1.5% HCl, 3 MPa	HPLC, UV-vis		[15]
Curculigo latifolia	root	TP, TF, pomiferin, etc.	antioxidant (DPPH, ABTS, TEAC)	100–200 °C, 10 MPa 30–120 min, 0.5 mL/min	LC-MS, UV		[16]
Citrus unshiu	peels	hesperidin and narirutin		110–190 °C 3–15 min	HPLC		[17]

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Glycine max	okara	genistin, daidzin, genistein, daidzein		100–200 °C, 5 min, 2–5 MPa, 10–30 g/mL	HPLC	Soxhlet (methanol, 108%)	[18]
onion	skins	quercetin, quercetin-4′-glucoside		100–190°C, 5–30 min, 9–13 MPa	HPLC	convention (methanol, 92.8%)	[19]
Puerariae lobata	root	puerarin, daidzin, daidzein 3-methoxypuerarin		100–200 °C, 15–75 min 1:10–1:25 g/mL	HPLC	reflux (ethanol 91.6%), UAE (water 95.9%)	[20]
Coriandrum sativum	seeds	TP, TF	antioxidant (DPPH)	100–200 °C, 10–30 min 3–9 MPa	UV		[21]
Citrus unshiu	peels	flavanones, polymethoxy-Flavones, etc.	anticancer, cardioprotectives	120–180 °C, 1.0–2.0 mL/min, 5.0 ± 0.1 MPa	GC, HPLC,	convention (methanol 75.0%; ethanol 41.6%; acetone 17.2%)	[22]
Phlomis umbrosa	whole part	TP, TF, iridoids glycosides	antioxidant (DPPH, ABTS)	110–200 °C, 10 MPa, 1–25 min	HPLC, ESI-MS	convention (ethanol; methanol; water)	[23]
Actinidia deliciosa	peels	TP, TF,	antioxidant (DPPH, ABTS, FRAP)	120–160 °C, 0–30 min, 3 MPa, pH 2–5.5	UV-vis, pH	convention (ethanol 81.9%)	[24]
Scutellaria baicalensis	root	baicalin, baicalein, wogonin, wogonoside		110–160 °C, 10–90 min, 20–100 mesh	HPLC	HRE (ethanol 93.0%)	[25]
Citrus unshiu	pomaces	TP, polymethoxylated flavones, sinensetin, etc.	antioxidant (DPPH, TP)	25–250 °C, 10–60 min, 0.1–5.0 MPa	HPLC, UV		[26]
citrus unshiu	peels	hesperidin, narirutin, prunin, naringenin, sinensetin, etc.	antioxidants (DPPH, FRAP), enzyme	145–175 °C, 15 min 5 MPa, 0.75–2.2 mL/min	HPLC	2M HCl extraction 42.9%; 2 M NaOH extraction 38.9%	[27]
citrus unshiu	peels	hesperidin and narirutin		110–200 °C, 5–20 min, 10 ± 1 MPa	HPLC, MS/MS	convention (ethanol 56.4%; methanol 35.8%; water 6.2%)	[28]
palatiferum Radlk.	leaves	TP, TF, protein, saponin, sugar, apigenin, kaempferol	antioxidants (DPPH, FRAP, ABTS),	110–270 °C, 15 min, 8 MPa 1:70 g/mL	HPLC, UV	convention (water 77.7%; methanol 32.8%), Soxhlet (ethanol 43.7%)	[29]
Glycyrrhiza uralensis Fisch.	root	TP, TF, liquiritin, flavanone, isoflavone	antioxidants (DPPH, ABTS)	80–320°C, 2–100 min, 7.0 MPa, 1:30 g/mL, pH 3–11	HPLC, MS/MS, UPLC	UAE (water 20.6%; ethanol 44.9%), MAE (water 25.6%; ethanol 63.8%)	[30]

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Tagetes erecta L.	flower residues	TP, TF, 5-HMF, reducing sugar, free amino acids	antioxidants (DPPH, ABTS)	80–260 °C, 15–90 min 1:20–1:60 g/mL,120 rpm	HPLC-DAD, UV	leaching (water 9.4%; methanol 69.9%; ethanol 68.8%; acetone 94.0%), UAE (water 9.9%; methanol 69.8%; ethanol 64.3%; acetone 87.6%)	[31]
Daucus carota	leaves	polyphenols, luteolin		110–230 °C, 0–114 min, 4 MPa	UV, PLC		[32]
Matricaria chamomilla L.	flowers	TP, TF, apigenin-7-O-glucoside, etc.	antimicrobial, cytotoxic activity	200 °C, 40 min, 1:50 g/mL	UHPLC, HESI-MS/MS, UV	Soxhlet (ethanol 129%), MAE (ethanol 117%), UAE (ethanol 104%)	[33]
Silybum murianum L	seeds	taxifolin, silychristin, silydianin, and silybin		75–250 °C, 40–60 min, 12.5 MPa, 0.1–0.5 mm	HPLC	convention (ethanol 101%; water 43.6%)	[34]
Echinacea purpurea L.	flowers	TP, TF	antioxidant (TEAC, ABTS)	103.4–216.56 °C, 3 MPa, 5.86–34.414 min	UV-vis		[35]
Humulus lupulus	pellets	TP, desmetylxanthohumol, prenylflavonoids, etc.	anti-inflammatory	50–200 °C, 30 min, 10 MPa	HPLC, MS/MS	convention (hexane 17.2%; ethanol 105%)	[36]
Kunzea ericoides	leaves	TP, TF, 5-HMF, quercetin, catechin, syringic acid, etc.	antioxidant (DPPH, FRAP)	150–210 °C, 0–40 min 15–35 g/mL, 4 MPa	HPLC, UV	convention (ethanol 37.5%)	[37]
Pistacia atlantica subsp. *mutica*	hull	TP, kaffesaure, ethyl vanillin, flavanomarein, etc.	antioxidant (DPPH), reducing power	110–200 °C, 30–60 min, 10–50 g/mL	HPLC-DAD, UV	HWE (85 °C 42.8%)	[38]
Satureja hortensis L.	whole part	TP, TF, rosmarinic acid, rutin, quercetin, etc.	cytotoxic, antibacterial	140 °C, 30 min 4 MPa, 1:20 g/mL	HPLC-PDA, UV	maceration (ethanol 57.2%), Soxhlet (ethanol 18.4%), UAE (ethanol 69.2%), MAE (ethanol 81.3%)	[39]
Urtica dioica L.	leaves	TP, TF, twenty-seven compounds	cytotoxic, antifungal, antimicrobial	125 °C, 30 min, 3.5 MPa, 1:30 g/mL	UHPLC-HESI-MS/MS	UAE (water 48.5%), MAE (water 100%)	[40]
Chamomilla recutita R.	flowers	2 flavonoids, 4 esters, 1 amino acid, 11 phenols, etc.		150 or 200 °C, 5.0 ± 0.1 MPa, 1.7 mL/min, 40 min	UV, HPLC, GC-MS		[41]
Glycine max	okara	TP, gallic acid, syringic acid, ferruric acid, etc.	antioxidant (ABTS, DPPH, FRAP)	150 °C, 4 MPa, 5–275 min 20 mg/mL	UV, HPLC		[42]

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Carménère grape	pomace	flavanols, stilbenes, and phenolic acids		90–150 °C, 5 min, 10 MPa, 15–50% glycerol	UPLC-MS		[43]
Zingiber officinale	root	TP, TF, four macro- and five microelements	antioxidant (OH·, ABTS, TRP, etc.)	80–180 °C, 1 h, 5MPa, 1:10 g/mL	UV-vis, ICP-MS	convention (water, 62.5%)	[44]
Momordica foetida	leaves	quercetin, kaempferol, isorhamnetin		100–300 °C, 5 mL/s 6.9± 1.4 MPa psi	UHPLC-q-TOF-MS		[45]

Ko and coworkers have investigated the relationship between flavonoid structure and SBWE. They found that flavonoids with an OH side chain were optimally extracted at lower temperatures than O-CH_3 and H side chains. The optimal temperatures of the glycoside forms are lower than that of the less polar aglycones [6]. Turner et al. found that different glycosidic compounds may be converted by their respective aglycones in less than 10 min reaction time in water from onion waste [46]. Similar results were obtained by Nkurunziza et al. [18] and Zhang et al. [20] who investigated the extraction of four flavonoids from okara and from *Puerariae lobata*, respectively.

2.2. Polyphenols

Polyphenols, also known as polyhydroxyphenols, are a structural class that is mainly natural, by the presence of more than one phenolic unit and being deprived of nitrogen-based functions. Many fruits, vegetables, herbs, tea leaves, nuts, and algae contain high levels of naturally occurring phenols. It has been reported that polyphenols can resist oxidation [47]. As shown in **Table 2**, extractions of polyphenols can be carried out either using a sole solvent such as water, methanol, ethanol or a mixture of solvents such as ethanol-water and methanol-water-formic acid.

Table 2. Subcritical water extraction of polyphenols.

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Allium ursinum L.	leaves	TP, TF, 5-HMF, catechin, p-cumaric, ferulic acids, etc.	antioxidant (DPPH, ABTS), Millard products	120–200 °C, 10–30 min, 0–1.5% HCl, 1:10 g/mL	HPLC-DAD		[47]
Terminalia chebula	fruits	TP, allic acid, corilagin ellagic acid	antioxidant (ABTS)	120–220 °C, 2–4 mL/min, 4 MPa	TLC, UV, MS, NMR, HPLC	Soxhlet (water 74.5%; ethanol 46.3%), HWE (water 46.3%)	[48]
Lycium ruthenicum Murr.	fruits	total anthocyanin, seven anthocyanins	antioxidant (ABTS, DPPH)	110–170 °C, 30–90 min, 1–3 min/L	HPLC, UPLC-MS	UAE (water 59.8%; methanol 81.1%)	[49]
Punica granatum L.	peels	TP, TF, punicalin, etc.		100–220 °C, 5–30 min, 3.0 MPa	UV-vis, HPLC	MAE (water 121%; ethanol 146%)	[50]
Castanea sativa	shells	tannins, phenolic acids, flavonoids, anthocyanins	antioxidant (DPPH, FRAP, ABTS)	51–249 °C, 6–30 min	UV-vis, LC/ESI-MS		[51]

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Salvia officinalis L.	by–products	TP, TF	antioxidant (DPPH, TEAC, reducing power)	120–220°C, 10–30 min, 3 MPa, 0–1.5% HCl	UV	maceration (water 59.9%)	[52]
Pistacia vera L.	hulls	gallotannin, anacardic acid, etc.	antioxidant (ABTS, FRAP)	110–190 °C, 6.9 MPa, 4 mL/min	HPLC-ESI/MS^n	UAE (methanol 83.9%))	[53]
Zingiber officinale	pulp and peel	6-gingerol, 6-shogaol	antioxidant (FRAP)	10 MPa, 110–190 °C, 5–40 min	HPLC	convention (methanol 114%; water 77.1%)	[54]
Sorfhum bicolor L.	bran	TP, oligomeric procyanidins, taxifolin, taxifolin hexoside	antioxidant (DPPH, ABTS), antiproliferative	110–190 °C, 5–40 min, 1:10–1:50 g/mL	HPLC, ESI-MS/MS	heating (water 74.9%)	[55]
Nelumbo nucifera	seed epicarp	TP, proanthocyanidin dimers, trimer, cyanidin, etc.	antiproliferation effect (MTT)	100–180 °C, 5–25 min, 1:20–1:60 g/mL, 1–5‰ $NaHSO_3$	HPLC-ESI-MS, UV	HWE (water 33.9%)	[56]
German chamomile	flowers	9 phenolic acids and derivatives	antioxidant, cytotoxic, enzyme	100 °C, 1–9 MPa, 30 min	UHPLC-DAD, MS/MS		[57]
Fagopyrum tataricum	grains	phenols, 13 phenolics, 4 flavonoids, 3 anthocyanins	antioxidant (TEAC, CAA and FRAP), cytotoxicity	220 °C, 60 min, 5 MPa, 1:60 g/mL	HPLC-MS, UV	UAE (water 83.5%)	[58]
A. uva–ursi	herbal dust	TP, TF	antioxidant (DPPH, reducing power)	120–220 °C,3 MPa, 10–30 min, 0–1.5% HCl	UV	maceration (water 38.5%; ethanol 69.5%)	[59]
Hippophaë rhamnoides L.	seed residue	TP, TF, proanthocyanidins	antioxidant (DPPH)	80–180 °C, 15–90 min, 1:10–1:50 g/mL, 6 MPa	UV	convention (water 19.6%; methanol 104%; ethanol 80.0%)	[60]
grape (Croatina)	pomace	TP, TF	antioxidant (DPPH)	100–140 °C, 8–15 MPa, 1–2 mL/min	UV	convention (water 5.3%; ethanol 7.87%)	[61]
Matricaria chamomilla L.	flowers	polyphenolic compounds, etc.	antioxidant, cytotoxic, enzyme inhibitory	65–210 °C, 30 min, 4.5 MPa	UHPLC-ESI-MS/MS, UV		[62]
Nelumbo nucifera	seedpods	TP, TF, proanthocyanidin dimer, isoquercetin, etc.	antioxidant, antiproliferative (HepG2)	100–180 °C, 30–70 mL/g, 5–25 min, 1–6‰ $NaHSO_3$	UV-Vis, HPLC, ESI-MS^n	HWE (water 91.4%)	[63]
Vitis vinifera L.	grape pomace	catechins, flavonols, tannins, proanthocyanidins, etc.	antioxidant (DPPH, ABTS)	40–120 °C, 10 min, 10.34 MPa, 10–40% NADES	UV, HPLC-ESI-MS		[64]

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
sweet chestnut	bark	TP, tannins, ellagic and gallic acids, ellagitannins, etc.	antioxidant (DPPH)	150–250 °C, 10–60 min, 10–30 mL/g, 4.5 MPa	UV-Vis, HPLC		[65]
Symphytum officinale	root	TP, TF	antioxidant (DPPH), enzyme inhibitory	120–200 °C,10–30 min, 0–1.5% HCl	UV, ELISA	UAE (methanol 2.5%; ethanol 17.4%); maceration (methanol 4.4%; ethanol 29.8%)	[66]
Pinot Nero	grape skins	TP		80–120 °C, 2 h,10 MPa, 2–5 mL/min	UV-Vis		[67]
Coffea arabica L.	spent coffee grounds	TP, caffeoylquinic acid, feruloylquinic acid, etc.	antioxidant (DPPH, ABTS)	160–180 °C, 35–55 min, 14.1–26.3 g/L	HPLC-$ABTS^{+}$, MS, UV		[68]
Curcumalonga L.	rhizomes	curcumin, demethoxycurcumin		120–160 °C, 6–22 min, 1–2.5 MPa	HPLC-UV, SEM		[69]
Curcuma longa L.	rhizomes	α-phellandrene, curcumin, β-caryophyllene, trans-β-farnesene, β-bisabolene, γ-curcumin, etc.		90–150 °C, 1–4 mL/min, 2 MPa, 0.5–1.5 mm	GC/GC-MS, GC -FID	HD (80.7%), Soxhlet (*n*-hexane 1.2-fold)	[70]
Curcuma longa L.	rhizomes	curcumin, demethoxycurcumin, bisdemethoxycurcumin		110–150 °C, 1–10 min, 0.5–10 MPa	HPLC	convention (ethanol, 1.13-fold)	[71]
Curcuma longa L.	rhizomes	curcumin, demethoxycurcumin, bisdemethoxycurcumin		90–250 °C, pH 1.0–5.5 5.0 MPa, 0.5 mL/min	HPLC, UPLC, LC-MS	Soxhlet (acetone, 1.17-fold)	[72]

2.3. Organic Acids

In general, organic acids in natural products are widely distributed in the leaves, roots, and fruits of the plants. The synthetic organic acids through chemical synthesis, enzymatic catalysis, and microbial fermentation are not discussed in this review. Organic acids are mostly soluble in water or ethanol and exhibit acidic properties, but they are difficult to dissolve in other organic solvents. It is generally believed that aliphatic organic acids have no special biological activity, but some natural organic acids such as citric acid, malic acid, tartaric acid, ascorbic acid, etc. have antibacterial, anti-inflammatory, hypoglycemic, antioxidant, and immune regulation effects. Depending on the organic acid in free state or in salt form, the extraction solvents could be water, dilute alkaline solution, diethyl ether, petroleum ether and cyclohexane, and other lipophilic organic solvents. A summary of recent studies on SBWE of organic acids are shown in **Table 3**.

Table 3. Subcritical water extraction of organic acids.

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Panax ginseng Meyer	root	TP, chlorogenic acid, caffeic acid, gallic acid, etc.	antioxidant (DPPH, ABTS, FRAP, HRS)	100–240 °C, 15 min, 4–9 MPa, 200 rpm	HPLC, UV		[73]
Helicteres isora L.		hexadecanoic acid, octadecnoic acid, heptadecen-8-carbonic acid etc.	antibiofilm, antioxidant, antimicrobial, antienzymatic	160 °C, 30 min, 1 MPa, 1: 30 g/mL	GC-MS, UV		[74]
XiLan olive fruit	olive dreg	TP, chlorogenic acid, gallic acid, syringic acid, etc.	antioxidant (ABTS, DPPH, reducing power)	100–180 °C, 5–60 min, 1:20–1:60 g/mL	LC-MS-IT-TOF, UV	convention (methanol 3.2%; ethanol 0.6%; DMK 0.9%)	[75]
Camellia oleifera Abel.	seeds	free fatty acids (palmitic acid, stearate, oleic acid, etc.), tea saponin	antioxidant (DPPH)	60–160 °C,2–7 MPa, 5–60 min, 1:3–1:25 g/mL	GC-MS, FT-IR	Soxhlet (petroleum ether 100%), cold pressed (100%)	[76]
sunflower seeds (Natura)	dehulled seeds	total proteins, total carbohydrates, TP	antioxidant capacities	60–160 °C, 5–120 min, 3 MPa, 1:10–1:30 g/mL	GC-FID, UV-Vis, HPLC	Soxhlet (hexane 67.3%)	[77]
cottonseed (Egypt)	cottonseed	linoleic acid, palmatic acid, oleic acid, myristic acid		180–280 °C, 5–60 min, 1:2–2:1 g/mL	GC-FID,	heating (hexane 89.5%)	[78]
green coffee (Robusta Uganda)	beans	chlorogenic acid		130–170 °C, 40–90 min, 0–30 % ethanol	HPLC	convention (ethanol 66.7%)	[79]
Nannochloropsis gaditana		fatty acids, omega-3, omega-6, lipid		156.1–273.9 °C, 6.6–23.4 min, 33–117 g/L	GC-FID, SEM	Soxhlet (n-hexane 100%)	[80]
Saccharina japonica		gallic, caffeic, vanillic, syringic, chlorogenic, p-hydroxybenzoic acids, etc.	antioxidant (DPPH, ABTS, total antioxidant (FRAP)	100–250 °C, 5 min, 5 MPa, 0.25–1.00 M ILs	HPLC, UV	convention (DMK 0.2%; DCM 0.3%; Et_2O 0.8%; IL 1.6%)	[81]
Haematococcus pluvialis		p-hydroxybenzoic acid, gallic acid, siringic acid, vanillic acid, etc.	antioxidant (ABTS, TEAC), antimicrobial activity	50–200 °C, 20 min, 10 MPa	HPLC-DAD-MS, SEM, GC-MS		[82]
Momordica charantia	fruits	TP, gallic acid, gentisic acid, chlorogenic acid	antioxidant (ABTS)	130–200 °C, 10 MPa, 2–5 mL/min	HPLC, UV	Soxhlet (methanol 4.9%), UAE (methanol 4.0%)	[83]
Morus nigra L., *Teucrium chamaedrys* L., *Geranium macrorrhizum* L., *Symphytum officinale* L.	leaves, flowers	TP, chlorogenic acid, gallic acid, vanillic acid, etc.	antioxidant, antifungal, antibacterial, cytotoxic	60–200 °C, 30 min, 1 MPa, 1:40g/mL	HPLC-DADUV		[84]

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Prunus avium L., *Prunus cerasus* L.	stems	3 alcohols, 10 organic acids, etc.	antioxidant, antiproliferative	150 °C, 30 min, 2 MPa	GC-MS, UV		[85]
Castanea sativa	nuts	ellagic acid, feru lic acid, gallic acid, etc.	antioxidant	120–135 °C, 15–60 min	HPLC		[86]
Solanum tuberosum	potato peel	TP, gallic acid, caffeic acid, chlorogenic acid, protocatechuic acid, etc.		100–240 °C, 30–120 min, 6 MPa	HPLC, UV	convention (methanol 1.6%; ethanol 2.0%)	[87]
Actinidia deliciosa	pomace	TP, chlorogenic acid, protocatechuic acid, etc.	antioxidant (DPPH, FRAP, ABTS)	170–225 °C 10–180 min, 5 MPa	UV, HPLC, pH		[88]
hypnea musciformis		chlorogenic, protocatechuic, and gallic acids, TP, TF, etc.	antioxidant (DPPH, ABTS), emulsify	120–270 °C, 10 min, 1:50–1:150 g/mL	pH, UV, HPLC		[89]
Carica papaya L.	papaya seeds	TP, 18 phenolic acids, 20 flavonoids, 1 stilbene, etc.	antioxidant (DPPH, β-carotene bleaching)	70–150 °C, 10 MPa, 1–40 min, 4 mL/min	LC-ESI-MS/MS, UV	Soxhlet (water 37.1%)	[90]
Zingiber officinale	ginger rhizome	12 sugars, 8 diols, 4 phenolic acids, etc.	antimicrobial, cytotoxic	150 °C, 1 h, 1:10 g/mL	HPLC-ESI-TOFMS	heating (water)	[91]
Chlorella sp. microalgae		TP, caffeic acid, ferulic acid, p-coumaric acid	antioxidant (DPPH)	100–250 °C, 5–20 min	UV, SEM, HPLC		[92]
Vitis vinifera	vine-canes	TP, flavonoids, phenolic acids, flavonols	antioxidant, antiradical	125–250 °C, 50 min	HPLC, UV		[93]
Cinnamomum Cassia Blume	cinnamon	coumarin, cinnamic acid, cinnamaldehyde, cinnamyl alcohol, etc.		110–130°C, 20–60 min, 2–4 MPa, 1:10 g/mL	HPLC		

The use of SBWE was explored for the extraction of gallic acid, chlorogenic acid, caffeic acid, ferulic acid, vanillic acid, and coumaric acid from various matrices. Inevitably, some other active components such as phenolics [73], flavonoids [45], proteins [73], lipids, peptides, amino acids, and other organic compounds were often coextracted. Švarc-Gajić et al. [85] have used SBWE for the extraction of alcohols, organic acids, sugars, and other organic compounds from both sweet and sour cherry stems, finding the chemical compositions of the two samples similar. Harun et al. [80] reported lipid extraction with a relatively high content of eicosapentaenoic acid from *Nannochloropsis gaditana*, finding 237 °C and 14 min to be the optimum extraction conditions.

2.4. Glycosides

Glycosides are compounds in which sugars or sugar derivatives are bound to another type of non-sugar substance (also called aglycones, ligands or substituents). Glycosides are linked by an O- N-, S-, or C-glycosidic bond between a sugar and a non-sugar component, which are widely found in the root, stems, leaves, flowers, and fruits of plants. Most glycosides are colored crystals, and generally a little bitter.

Glycosides extracted by SBWE have been proven to have antioxidant activities and tyrosinase inhibitory activity, as shown in **Table 4** [94][95][96][97][98][99][100][101][102][103]. Gao et al. [95] has performed SBWE of phenolic compounds from pomegranate seed residues at 80–280 °C. The results showed that TP increased with the rise of extraction temperature from 80 °C to 220 °C and decreased from 220 °C to 280 °C. At 80–220 °C, the breakage of the bonds led to the increase of TP, however, a higher temperature caused the phenolics to degrade. In addition, they compared SBWE with leaching

and UAE using water (room temperature) and organic solvents namely methanol, ethanol, and acetone. TP and antioxidant capacities of SBWE (120 °C) were not as high as organic solvents; however, with respect to the extraction time (2 h for leaching vs. 30 min for SBWE) and toxicity, subcritical water is more acceptable. Meng and Cheng [100] have studied 13 phenolic compounds and 20 inorganic elements of *Erigeron breviscapus*. They also have found similar results, as the glycosides are not stable at a high temperature and with a long extraction time. For example, scutellarein and apigenine are the aglycones of corresponding acutellarin and apigenin 7-glucuronide, when at high temperature glycosidic bonds become unstable and begin to decompose to its glycone and aglycone. Haznedaroglu et al. [98] have optimized the parameters such as temperature, extraction time, and flow rate. Temperature and extraction time were found as the most effective parameters for TP and total flavonoids while extraction time and flow rate for anthocyanin contents. In addition, temperature and time were the leading parameters for the effectiveness of extracts on tyrosinase inhibition.

Table 4. Subcritical water extraction of glycosides.

Samples	Medicinal Parts	Compounds Extracted	Extracts Activity	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Phaleria macrocarpa	**fruits**	**mangiferin**		**323–423 K, 1–7 h, 0.7–4.0 MPa**	**HPLC, LC-MS**	**convention (water 69.6%; ethanol 34.1%; methanol 108%), HRE (water 85.7%; ethanol 60.8%; methanol 115%), Soxhlet (water 86.1%; ethanol 55.8%; methanol 113% methanol)**	**[94]**
***Punica granatum* L.**	**pomegranate seed**	**TP, kaempferol -3-O-rutinoside**	**antioxidant (DPPH, ABTS)**	**80–280 °C, 5–120 min, 1:10–1:50 g/mL, 6.0 MPa**	**HPLC-DAD, UV, HPLC-ABTS$^+$**	**leaching (water 40.6%; methanol 79.7%; ethanol 41.7%; acetone 45.5%), UAE (water11.3%; methanol 20.6%; ethanol 18.9%; acetone 15.2%), Soxhlet (methanol 71.4%; acetone 39.7%)**	**[95]**
***Teucrium montanum* L.**	**aerial parts**	**rutin, naringin, epicatechin, etc.**	**antioxidant (DPPH, FRAP)**	**60–200 °C, 30 min, 1–10 MPa, 1:10 g/mL**	**HPLC-PDA, UV**		**[96]**
Paeonia lactiflora	**root**	**albiflorin, paeoniflorin**		**100–260 °C, 10–60 min,10–40 mL/g**	**HPLC**	**reflux (water 83.5%), UAE (ethanol 77.8%)**	**[97]**
***Morus nigra* L.**	**fruits**	**TP, TF, cyanidin 3-glucoside, etc.**	**40–80 °C, 20–60 min, 2–6 mL/min, 15 MPa**	**tyrosinase inhibitory activity**	**UPLC-DAD-ESI-MS/MS**	**shaker (ethanol:water 116%), UAE (ethanol:water:TFA 134%)**	**[98]**
Stevia rebaudiana	**leaves**	**TP, stevioside, rebaudioside A**	**antioxidants (DPPH)**	**100–150°C, 30–60 min, 23 MPa, 1:10 g/mL**	**HPLC-UV, UV**		**[99]**
Erigeron breviscapus	**whole parts**	**scutellarin, 20 inorganic elements, etc.**	**antioxidant (DPPH)**	**120–140 °C, 5–15 min, 150–420 um**	**HPLC, HPLC-MS**	**reflux (methanol 86.1%; ethanol 84.8%)**	**[100]**
***Mangifera indica* L.**	**leaves**	**quercetin3-d-glucoside, mangiferin**	**antioxidant (DPPH)**	**100 °C, 4 MPa, 10 g/min, 3 h**	**UV, HPLC**	**$SCCO_2$ (20% methanol 18.7%)**	**[101]**
***Crocus sativus* L.**	**stigmas**	**picrocrocin, safranal, crocin**		**5–15 min, 105–125 °C**	**GC-MS, UV, HPLC**		**[102]**
***Glycyrrhiza uralensis* Fisch**	**licorice root**	**TP, glycyrrhetic acid, glycyrrhizin, liquiritin**	**antioxidant (DPPH, reducing power)**	**50–300 °C, 10–60 min, 0.002–5 MPa**	**HPLC, UV-Vis**		**[103]**

2.5. Carbohydrates

Carbohydrates is a very common term that include sugars, starch, and cellulose, which are an important class of organic compounds widely distributed in nature. The saccharides are divided into four groups: monosaccharides, disaccharides, oligosaccharides, and polysaccharides. As shown in **Table 5**, carbohydrates extracted by SBWE possess antioxidants [104], antimitotic [105], and growth inhibitory effects [106].

Table 5. Subcritical water extraction of carbohydrates.

Samples	Medicinal Parts	Compounds Extracted	Activity/Mixtures	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Lycium barbarum	berries	total sugar content	antioxidant (FRAP, TEAC), immunomodulatory	1:30 g/mL, 110 °C, 5 MPa	HPGPC	HWE (water 71.5%), UAE (water 89.9%), UWE (water 132%)	[107]
sunflower	sunflower heads	galacturonic acid, pectin		10–50 min, 2–8 mL/g, 100–140 °C, 0.2–1 MPa	TG/TGA, DSC, UV−vis, FTIR, HPSEC, NMR		[108]
Aronia melanocarpa	chokeberry stems	1 amino acid, 8 alcohols, 11 sugars, 2 fatty acids, etc.	antioxidant (DPPH), enzyme inhibitory activity	130 °C, 3.5 MPa, 20 min, 1:20 g/mL	GC-MS		[109]
Lentinus edodes	fruit bodies	hetero–polysaccharides, xylose, mannose, etc.	antioxidant (OH·, DPPH, ABTS)	120–160 °C,30–50 min, 0.033–0.05 g/mL	UV-vis, SEM, GC, GPC, FT-IR		[110]
Lentinus edodes	fruit bodies	l-rhamnose, d-arabinose, d-xylose, d-mannose	antioxidant (ABTS), growth inhibitory effect	100–150 °C, 10–30 min, 5 MPa	FT-IR, UV-Vis, AFM, GC, HP SEC-MALLS		[106]
Lentinus edodes	fruit bodies	polysaccharides, rhamnose, arabinose, xylose, etc.	antioxidant (DPPH, reducing power)	140 °C, 40 min, 1:25 g/mL, 5 MPa	GC, FT-IR, AFM, SEM		[111]
Lentinula edodes	fruit bodies	TCC, total β-glucan, chitin	HMGCR, immuno-modulatory	200 °C, 11.7 MPa, 15–60 min	GC-MS, HPSEC, NMR	UAE (water 65.2%), HWE (water 32.3%), SPE (water 33.0%)	[112]
Grifola frondosa	fruit bodies	total polysaccharide, total protein	antioxidant (DPPH, reducing power)	100–230 °C, 2–4 min, 20–100 mesh, 5 MPa	FT-IR, SEM	HWE (water ~87.8%)	[113]
Sagittaria sagittifolia L.	fruit bodies	polysaccharides	antioxidant (DPPH, ABTS, reducing power)	150–190°C, 12–20 min, 1:20–1:40 g/mL, pH 7–9	FT-IR, 1H and 13C NMR, UV	HWE (water 55.8%)	[114]
Sagittaria sagittifolia L.	fruit bodies	l-rhamnose, d-arabinose, d-xylose, d-mannose	antioxidant, immuno-modulatory	170°C, 16 min	HPLC, GC, SEM, IR, AFM, HPSEC-MALLS	HWE (water 75.6%); UAE (water 96.1%)	[115]

Samples	Medicinal Parts	Compounds Extracted	Activity/Mixtures	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Sagittaria sagittifolia L.	fruit bodies	α-pyranose polysaccharide, β-pyranose polysaccharide	immuno-stimulatory	1 MPa, pH 7,170 °C, 16 min, 30:1 mL/g	IR, GC-FID, UV, HPSEC, AFM		[116]
Cordyceps militaris	fruit bodies	total sugars, protein and uronic acid		180 °C, 13 min, pH = 8, 21 mL/g	IR, GC, AFM, GPC-MALLS		[117]
wheat	bran	monosaccharide, etc.	antioxidants (DPPH)	160–180°C, 5–60 min	HPAEC-PAD, SEC		[104]
Saccharina japonica		fucoidan, fucose, glucose, galactose, mannose, etc.	antioxidant, antimitotic anti-proliferative	100–180 °C, 5–15 min, 2–8 MPa	FTIR, TGA, UV-Vis	convention (0.05 M HCl 100%)	[105]
Citrus grandis L.	pomelo peel	pectin		90–120 °C, 3–10 MPa	HPSEC-MALLS		[118]
Theobroma cacao L.	cacao pod husks	xylose, arabinose, etc.		121 °C, 30 min, 10.3 MPa	FT-IR, GC-FID, SEM	convention (4% citric acid 76.1%)	[119]
Kappaphycus alvarezii. A		κ-carrageenan, glucose, 3,6-anhydrogalactose, etc.	antioxidant (DPPH, ABTS)	60–180°C, 5 MPa, 5 min	FTIR, TGA, XRD	convention (water 94.3%; water with IL 101%)	[120]
Pseuderanthemum palatiferum	leaves	TCC, monosaccharides	anticoagulant, antioxidant	150–200°C, 5–10 mL/min	HPLC, GPC, NMR, UV	convention (0.1 M NaOH 48.8%)	[121]
wheat	bran	TCC, reducing sugar, arabinose, xylose, etc.	antioxidant, α-amylase inhibitory	140 °C, 5 MPa, 30 min	SEC-MALLS, FT-IR, DLS, DSC, UV	SBWE (water with citric acid 97.6%); UWE (water with citric acid 103%)	[122]
Lycium barbarum L.	fruits	polysaccharides	antioxidant (O2·, OH·, DPPH)	5 MPa, 25 mL/g, 110 °C, 1 h	UV	HWE (water 86.2%); UAE (water74.9%); UWE (water 111%)	[123]
Cocos nucifera L.	defatted coconut	mannose, galactosamine, xylose, rhamnose, etc.	antioxidant, hypoglycaemic, adsorption	1:10–1:50 g/mL, 10–50 min, 120–200 °C, 20–100 mesh	HPLC, XRD, TGA, DTGA, SEM, FT-IR		[124]
okara		polysaccharides, TP, TF	antioxidant (ABTS, DPPH)	1:30 g/mL, 160–230 °C, 10 min	UV		[125]
Saccharina japonica		polysaccharide, fucoidan, alginate	antioxidant (ABTS, DPPH, FRAP)	100 –150 °C, 1–5 MPa, 1:30–1:50 g/mL	IR, DSC, TGA, ^{1}HNMR, HPLC, HPSEC-ELSD		[126]
Passiflora edulis	fruit peel	pectic polysaccharide, mannose, glucose, etc.	antioxidant (DPPH)	100–160 °C, 5.64–7.94 min, 10–30% ethanol	HPLC, UV, viscometer		[127]

Samples	Medicinal Parts	Compounds Extracted	Activity/Mixtures	Extraction Conditions	Analytical Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Chlorella vulgaris, Sargassum vulgare, Sargassum muticum, Porphyra spp., *Cystoseira abies–marina, Undaria pinnatifida and Halopitys incurvus, Rosmarinus officinalis* L., *Thymus vulgaris, Verbena officinalis*	microalgae, algae, leaves	sugar, TP, melanoidins	antioxidant (ABTS, O_2^-)	100–200 °C, 20 min, 10 MPa	UV		[128]
rice bran	bran	protein, TCC, TP	antioxidant (DPPH)	120–250 °C,0.5–5 mL/min	UV, UV-Vis		[129]
Nizamuddinia zanardinii		TCC, rhamnose, xylose, arabinose, fucoidan, fucose	antioxidant, anticancer, macrophage, etc.	425 rpm, 10–30 min, 90–150 °C, 0–40 mL/g, 0.75 MPa, 1500 W	FT-IR, GC-MS, SEM, UV, HPSEC-MALLS-RI		[130]
Dendrobiumnobile Lindl.	stems	polysaccharide, arabinose, galactose, glucose, etc.	antioxidant (OH·, ABTS)	0.5–1.5 MPa, 5–20 min 120–160 °C, 1:25 g/mL	UV−vis, GPC, HPLC, HPAEC		[131]
Ecklonia maxima	TP	polysaccharide, sulphate, and alginate	antioxidant (ABTS)	100–180 °C, 5–30 min, 10–50 mL/g, 4 MPa	UV, elemental analysis, ICP-MS	convention (70% ethanol 0%; 0.05 M HCl 20.1%)	[132]
Vitis vinifera	grape pomace	glucose, fructose, galactose, arabinose, mannose, etc.	antimicrobial, antioxidant (DPPH)	170–210 °C, 10 MPa, 5–10 mL/min	HPLC, UV		[133]
green coffee beans	spent coffee grounds	carbohydrates, phenolics	antioxidant, antibacterial	150–220 °C, 7 MPa, 10 mL/min,	HPLC, UV		[134]
Tamarindus indica	seed	TP, xyloglucan	antioxidant (DPPH)	100–200°C, 1:20 g/mL	SEC, UV	convention (water 74.6%)	[135]
Mentha arvensis	leaves	carbohydrates, apocynin	antioxidant (DPPH)	180–260 °C, 1:20 g/mL, 5 min	HPLC, GC-MS, UV,		[136]

2.6. Essential Oils, Alkaloids, Quinones, Terpenes, Lignans, and Steroids

Subcritical water has also bee used to extract essential oils, alkaloids, quinones, terpenes, lignans, and steroids from plant and other materials. A summary of SBWE of essential oils, alkaloids, quinones, terpenes, lignans, and steroids can be found in Table 6.

Table 6. SBWE of essential oils, alkaloids, quinones, terpenes, lignans, and steroids.

Samples	Medicinal parts	Compounds Extracted	Extraction Conditions	Methods	Other Extraction Methods (Solvent, Ratios of Yields)	Ref.
Essential oils						
Thymbra spicata L.	leaves	α-thujene, α-pinene, terpinen-4-ol, p-cymene, γ-terpinene, 1-carvone, thymol, carvacrol, etc.	100–175 °C, 1–3 mL/min, 2–9 MPa, 30 min	GC-TOF/MS, GC-FID		[12]

Aquilaria malaccensis	leaves	butanal, cyclopentanone, acetoxyacetone, benzaldehyde, acetophenone, creosol,etc.	100–271 °C, 1–34 min, 0.08–0.22 g/mL	GC-MS, SEM, FT-IR	HD (95.4%)	[22]
Mentha piperita L.	peppermint leaves	TP, menthone, menthol, eriocitrin, etc.	40–160 °C, 10.3 MPa, 1–30 min	GC-MS, FID, HPLC	convention (methanol 53.2%)	[28]
Coriandrum sativum L.	coriander seeds	thujene, sabinene, pinene, myrcene, cymene, limonene, ocimene, terpinene, terpinolene, etc.	100–175 °C, 1–4 mL/min, 0.25–1 mm, 2 MPa, 20 min	GC-FID, GC-MS	HD (1.54-fold), Soxhlet (hexane 1.4-fold)	[174]
Coriandrum sativum L.	coriander seeds	3,4-dimethoxycinnamic acid, coumaric acid, sinapic acid,cis-and trans-linalooloxides, linalool, etc.	100–200°C, 10–30 min, 3–9 MPa	HPLC-MS/MS, GC-MS		[175]
Kaempferia galangal L.	rhizome	ethyl-p-methoxycinnamate, d-limonene, eucalyptol, tridecane, camphor, borneol, tetradecane, etc.	120 °C, 10 MPa, 30 min	GC-MS	HD (82.3%), UWE (100%)	[176]
Piper betle	leaves	4-allyl resorcinol, chavibetol	2 MPa, 10–90 min, 50–250 °C, 0.25–1 mm, 1–4 mL/min	HPLC-UV	convention (water 92.2–111%; methanol 96.6–110 %)	[177]
Aquilaria malaccensis	leaves	nonacosane, triacontane, pentadecanal, 9-octadecenal, (Z)-, tetradecanal, tetrapentacontane, guaiacol	100–271 °C, 1–34 min	GC/MS, SEM, BET		[178]
laurel	leaves	α-phellandrene, β-pinene, 1,8-cineole, borneol, nona-3,7-dienol, isobornyl acetate, γ-terpineol, etc.	15 min, 50–200 °C, 1.5–15 MPa, 0.5–5.0 mL/min	GC-MS, GC-FID		[179]

Citrus hystrix	leaves	linalool, isopulegol, neoisopulegol, citronellal, 4-terpineol, citronellol, geraniol, menthoglycol, etc.	120–180 °C, 5–20 g/mL, 5–30 min	GC-MS	HD (28.2%)	[180]
Coriandrum sativum L.	coriander seeds	α-pinene, β-pinene, camphor, methylchavicol, γ-terpinene, linalool, geraniol, carvacrol, etc.	100–200 °C, 1:10 g/mL, 2 MPa, 20 min	GC-MS, GC-FID	HD (27.0%), Soxhlet (DCM 6.5-fold), $SCCO_2$ (4-fold)	[181]
Lavandula L.	lavender flowers	a-thujene, a-pinene, camphene, sabinene, pinene, myrcene, hexylacetate, terpinene, limonene, etc.	125 °C, 3 MPa, 30 min	GC-MS, FID	HD (1.2-fold), US-HD (1.3-fold), NaCl-HD (1.3-fold)	[182]
		Alkaloids				
Sophora Ait.	root	cytisine, matrine, sophoridine, sophocarpine, oxymatrine	70–190 °C, 5–14 min, 4.0–13.8 MPa	CE	ASE (ethanol 78.1%)	[16]
black tea brick	leaves	theophylline, epicatechin gallate, caffeine, etc.	120–180 °C, 7–42 min, 6–18 mL/min	HPLC		[46]
Symphytum officinale L.	root	lycopsamine, echimidine, lasiocarpine, symviridine	60–120 °C, 40 min	HPLC, LC-MS, MS^n	HRE (methanol 2.8-fold)	[183]
hydrastis canadensis	root	hydrastine, berberine	100–160 °C,1–10 MPa, 5–60 min, 0.5–1.5 mL/min	HPLC-DAD	reflux (methanol 90.8%), UAE (methanol 106%)	[184]
cocoa	shells	TP, theobromine, theophylline, caffeine, epicatechin, etc.	120–220 °C, 15–75 min, 1:10–1:30 g/mL	HPLC, UV		[185]

Musaceae, *Beta vulgaris*	peels	dopamine, total betacyanin, betaxanthin	150°C, 5 min, 3 MPa, 1:20 g/mL	HPLC, UV-Vis	infusion (100%), decoction (1.2-fold), maceration (97.4%), UAE (101%), MAE (50.3%)	[186]
Coffeea arabica, C. arabica, C. canephora var. robusta, C. canephora var. robusta	coffee silver skin	total sugar, reducing sugar, protein, TP, caffeine, HMF, etc.	180–270 °C, 10 min, 1.0–5.3 MPa	HPLC, UV	convention (0.1 M HCl 96.6%; 0.1 M NaOH 1.5-fold)	[187]
Quinones						
Rheum tanguticum	root	damnacanthal	33–67 min, 100–200°C, 1.4–4.6 mL/min,	HPLC, NMR, HSCCC		[17]
Garcinia mangostana Linn	mangosteen pericarps	TP, xanthone	120–160 °C, 1–10 MPa，5–60 min, 10–30% DES	UV-vis, FT-IR, SEM		[188]
Phaleria macrocarpa	mahkota dewa fruits	mangiferin	4.0 MPa, 5 h, 50–150 °C	HPLC		[189]
Lithospermum erythrorhizon	root	shikonin, acetylshikonin, β-dimethylacrylshikonin, etc.	40–60 mesh, 120 °C, 5 MPa	UV, HPLC-ELSD	$SCCO_2$ (86.3%), Soxhlet (ethyl acetate 95.4%), UWE (1.4-fold)	[190]
Morinda citrifolia	root	alizarin	4 MPa, 150 and 220 °C, 1.6–4 mL/min	RP-HPLC-UV		[191]
Morinda citrifolia	root	1,2-dihydroxyanthraquinone, alizarin	110–220 °C, 2–6 mL/min	UV-Vis	ethanol (3 d)	[192]

Morinda citrifolia	root		4 MPa, 150–200 °C, 2–6 mL/min	UV-Vis	convention (ethanol 81.16%), Soxhlet (ethanol 97.94%), UAE (ethanol 79.62%) SWBE (96.41%)	[193]
		Terpenes				
Hedyotis diffusa Willd.	whole plants	ursolic acid	120–200 °C, 10–50 min, 20–40 mL/g, 0.6–3.0 MPa	HPLC-ESI-TOF-MS	maceration (ethanol 58.8%), HRE (ethanol 78.4%), UAE (ethanol 90.4%), MAE (ethanol 74.9%)	[13]
Centella asiatica	whole plants	asiatic acid, asiaticoside	100–250°C, 10–40 MPa, 5h	HPLC, DLS		[14]
basil, oregano	leaves	limonene, citronellol, etc.	100 and 150 °C, 10 min	GC-FID		[48]
Ganoderma lucidum	fruits	ganodermanon-triol, ganoderic acids, lucidumol	100–200 °C, 5–10 MPa, 5–60 min	HPLC, SEC-UV, SEM, MALDI-TOF		[194]
Orostachys japonicus	stems, leaves	triterpene, camellia, etc.	110–260 °C, 5–20 min, 10 MPa	HPLC-MS		[195]
Betula pendula	birch bark	betulinic acid	160–200 °C, 10–30min, 10 MPa	HPLC		[196]

Inula racemose	plants	igalan, soalantolactone, alantolactone	23.2–56.8 min, 1.3–4.7 mL/min, 129.5–230.5 °C	HPLC, ^{1}H-NMR ^{13}C-NMR, MS	Soxhlet (ethanol 100%), UAE (ethanol 70.36%), $SCCO_2$ (76.06%)	[197]
Semen richonsanthis	seeds	3,29-dibenzoylkarounidiol, polysaccharides	80–160 °C, 5.0–30.0 min	HPLC, UV, SEM		[198]
Cucurbita pepo	pumpkin peel	14 carotenoid compounds	120 °C, 3 h, 5 MPa	UV, HPLC	$SCCO_2$ (75.4%)	[199]
Betula pendula	birch bark	sesquiterpenes, steroids	10 min, 100–200 °C	LC, GC/MS, NMR		[200]
S. rebaudiana	Bertoni leaves	steviol glycosides, tannins, chlorophyll A	100–160 °C, 5–10 min, 10.34 MPa, 1:3 g/mL	HPLC, UV, UV/Vis		[201]
		Lignans				
Linum usitatissimum L.	flaxseed	SDG lignan, phenolics, flavonoids	160–180 °C, 5–60 min, 10 MPa	HPLC-MS/MS, UV		[41]
Sesamum indicum L.	sesame seeds	lignans, TP, flavonoids, flavonols	140–220 °C, 8–14 MPa, 0–95% ethanol, 0–75 min	UV		[42]
Linum usitatissimum L.	flaxseed	total fat content, SDG lignan	120–180 °C, 15–90 min, 10–13.8 MPa	HPLC-MS/MS, UV		[43]
Sinopodophyllum hexandrum	root	podophyllotoxin	12 mL/g, 3 MPa, 2ml/min, 120–240 °C	HPLC		[202]
		Steroids				
Pfaffia glomerata, Amaranthaceae	ginseng root	sugar, fructooligosaccharides, beta-ecdysone	80–180 °C, 5–15 min, 2–12 MPa	HPLC-ELSD, HPLC		[24]

Panax ginseng C.A. Meyer	ginseng root	TP, maltol, panaxadiol, panaxatriol	150–200 °C, 5–30 min, 100 MPa	HPLC, UV	convention (water 32.6%; methanol 24.1%; ethanol 18.7%)	[25]
Panax ginseng C.A. Meyer	ginseng root	total ginsenosides, total sugar, 1-oleanane ginsenosides, etc.	120–200 °C, 20 min, 1:20 g/mL, 6.0 MPa	FT-IR, UV, UFLC-MS/MS	heating (water, 30.9%; ethanol 94.4%)	[26]
grapevine	root, wood, cane	E-piceid, E-piceatannol, E-resveratrol, E-parthenocissin, etc.	100–190 °C, 5–30 min, 10 MPa	LC-DAD/ESI-IT, Q-TOF, NMR	ASE (116% for cane; 103% for wood; 1.5-fold for root)	[203]
Withania somnifera L	root leaves	TP, withanoside IV V, withaferin A, withanolide A, B	100–200 °C, 10–30 min, 10 MPa	HPLC, UV	maceration (water 31.7%), Soxhlet (ethanol 39.2%), MAE (methanol 45.8%)	[204]
Acanthophyllum glandulosum	root	saponin	121 °C, 0.15MPa, 15 min, pH 4–9	FT-IR, UV-vis, HPLC		[205]
Vaccaria segetalis	cowcock seed	vaccarosides, segetosides	125–175 °C, 15–180 min		USE (methanol 46.8%; water 27.9%; ethanol 5.2%)	[206]

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