

Review

## Innovations and modifications of current extraction methods and techniques of citrus essential oils: a review

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Received: 20 May 2024 / Accepted: 16 July 2024

Published online: 27 August 2024

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### Abstract

The genus Citrus of the Rutaceae family remains one of the beneficial fruit crops that produce high quantities of essential oils that have pharmaceutical, biological, and food preservative applications. Despite the numerous benefits of citrus essential oils (CEOs), there is a major challenge in choosing the most efficient extraction method(s) for large-scale production of quality CEOs to meet industrial, research, and domestic demands. This review provides a general overview of the listed citrus species, the chemical composition of their essential oils, medicinal uses, and the major methods of extraction of citrus essential oils from 10 selected citrus species. A meticulous, in-depth review of the various methods of CEOs extraction has been provided, along with their advantages, limitations, and novel modifications. This comprehensive literature review expounded on the current extraction methods for citrus essential oils and the various modifications developed to reduce the extraction time, excessive energy consumption, CO<sub>2</sub> production, and quality, as well as to improve the extraction yield.

### Article Highlights

- General overview of ten (10) selected citrus species and their essential oil composition and applications.
- The advantages and disadvantages of various methods of extraction of citrus essential oils for research and industrial applications.
- Innovative modifications of the conventional and advanced methods for extraction of citrus essential oils have better efficiency.

### Abbreviations

5-MOP-5	Methoxy psoralen
ATR-FTIR	Attenuated total reflection fourier-transform infrared spectroscopy
CEOs	Citrus essential oils
DRIFT	Diffuse reflectance infrared Fourier transform
EOs	Essential oils
FESEM	Field emission scanning electron microscopy
HD	Hydro distillation
HPLC	High-performance thin-layer chromatography

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HPLC-ToF-MS	High-performance thin-layer chromatography time of flight mass spectrometry
HPTLC	High-performance thin-layer chromatography
HSCC	High-speed counter-current chromatography
GC-MS	Gas chromatography-mass spectrometry
GC-C-IRMS	Chromatography-combustion-isotope ratio mass spectrometry
GC-FID	Gas chromatography with flame-ionization detection
GC-O	Gas chromatography olfactometry
LDL	Low-density lipoprotein
MAE-IL	Ionic liquid-based microwave-assisted extraction
MASD	Microwave assisted steam distillation
MASE	Microwave-assisted solvent extraction
MDGC-MS	Multi-dimensional gas chromatography-mass spectrometry
MHG	Microwave hydro-diffusion and gravity
NMR	Nuclear magnetic resonance
RP-HPLC	Reversed phase high-performance thin-layer chromatography
SC-CO <sub>2</sub>	Supercritical carbon dioxide
SFE	Supercritical fluid extraction
SFME	Solvent-free microwave extraction
SD	Steam distillation
UAE	Ultrasound-assisted extraction
UHPLC-TOF-MS	Ultra-high performance liquid chromatography time-of-flight mass
UA-HS-SPME	Ultrasonic-assisted headspace solid phase micro-extraction

## 1 Introduction

The *Citrus* genus is one of the most popular members of the family Rutaceae. The *Citrus* genus is made up of 17 plant species, which are mostly trees or shrubs and are widely cultivated in tropical and subtropical regions of the world. Even though *Citrus* species are cultivated in different parts of the world, their origin can be traced to Southern China, Korea, Japan, Northern Myanmar, and India [1, 2]. Citrus is one of the most cultivated fruit plants in the world, and its production was around 144 million metric tons in 2020 [3]. Between 50 and 60% of citrus fruits are consumed freshly, while the remainder is processed into different products by industries [4]. China, Brazil, the USA, India, and Mexico are the largest producers of citrus fruits. The common Citrus species grown for their fruits include sweet orange (*Citrus sinensis* Osbeck), key lime (*Citrus aurantifolia* (Christm.) Swingle), bergamot orange (*Citrus bergamia* Risso), bitter orange (*Citrus aurantium* L.), lemon (*Citrus limon* L.), mandarin orange (*Citrus reticulata* Blanco), grapefruit (*Citrus × paradisi*), kumquat (*Citrus japonica*), and yuzu (*Citrus junos* Siebold ex Tanaka). Citrus plants are mainly valued for fruits that are rich in ascorbic acid, flavonoids, and polyphenols, which have vital health benefits [5]. There is growing interest in the use of natural products in disease treatment, and several studies have highlighted the medicinal benefits of citrus species and their derived bioactive compounds. Citrus fruits, seeds, juices, and fruit peel/rind oils have been used in the formulation and preparation of most products, such as pharmaceuticals, food preservatives, cosmetics, detergents, air fresheners, deodorants, and sweet-scented perfumes [6, 7]. The fruit peels, leaves, aerials, seeds, and flowers of citrus plants contain volatile essential oils characterized by a sweet-scented aroma and possessing vital phytochemicals [8, 9]. Essential oils primarily contain a mixture of aromatic compounds stored in special parts of the plants, specifically in the oil glands of the fruit peels, leaves, seeds, and flowers [4]. These essential oils are endowed with several medicinal benefits, which include antimicrobial, aromatherapy, anti-inflammatory, anti-diabetic, anti-cancer, and insecticidal properties [10, 11]. The biological activities of Citrus essential oils are attributed to the presence of a mixture of complex compounds, mainly terpenes, monoterpenes, alcohols, aldehydes, esters, and ketones [12]. Most of the compounds are volatile and thus sensitive to conditions like high temperatures (heat), pressure, oxygenation, and light [13]. Also, the composition and quantities of these compounds can vary significantly in various citrus essential oils depending on the species, climatic conditions, geographical location, and methods of extraction and storage of the essential oils [14]. The yield, composition, and quality of the citrus essential oil differ significantly depending on the pre-extraction treatment and the choice of extraction method(s) [15]. The main challenge is to determine the efficient extraction method and the conditions under which the constituents of the essential oil reflect the actual composition and quality of the plant storage parts

[16]. Efficient methods of extraction are needed to preserve the vital constituents, increase the yield, and limit the variations in the components [17]. In this review, a brief description of 10 selected citrus species will be given, and the various methods of extraction and purification of their essential oils will be discussed extensively by comparing their efficiencies, disadvantages, innovations, and modifications.

## 2 Methodology

In this review, we reviewed general scientific articles or publications, conference papers, book chapters, and theses indexed in different databases, including, Scopus, PubMed, Wiley, Taylor & Francis, Springer, Google Scholar, Science Direct, and Web of Science. The review focused on 10 species of the Citrus genus by elucidating their general overview of the plants, biological applications, methods of extraction, and detection or quantification of their essential oils. The review covered essential oils extracted from the following species, lemon (*C. limon* L.), sweet orange (*Citrus sinensis* L. Osbeck), mandarin orange (*Citrus reticulata* Blanco), lime (*Citrus aurantifolia* (Christm.) Swingle), Bergamot orange (*Citrus bergamia* Risso), bitter orange (*Citrus aurantium* L.), mandarin coleopatra citrus (*Citrus reshni*), grapefruit (*Citrus paradisi*), Kumquat (*Citrus japonica*), and yuzu (*Citrus junos*). The search terms used were "citrus species," "citrus essential oil extraction," "essential oil extraction methods," "steam distillation," "cold pressing," "solvent extraction," "hydro-distillation," "microwave-assisted extraction," "supercritical fluid extraction," and "ultrasound-assisted extraction."

## 3 General overview of ten selected citrus species

### 3.1 Sweet orange (*Citrus sinensis* L. Osbeck)

Sweet orange, *Citrus sinensis* (L) Osbeck, is one of the widely cultivated citrus species with increased production in different parts of the world, mostly for the consumption of fruits due to its nutritional and flavoring value [18]. Apart from their nutritional value, they are also a rich source of essential oils with several applications in pharmaceuticals, food processing and storage, perfumery, and cosmetics [11]. Sweet orange (*C. sinensis*) is commonly cultivated in subtropical regions and produced in large quantities in different parts of the world. It accounts for approximately 70% of all yearly production of citrus species [19]. The annual production of *C. sinensis* is reported to be the highest among most fruits with approximately 146.5 million tons in 2017 [19–21] and is mainly produced by countries like Brazil, the USA, Mexico, China, and Spain [22]. *C. sinensis* essential oil serves as the dominant source of monoterpene hydrocarbons and appreciable levels of myrcene,  $\beta$ -pinene, and sabinene [23]. Other volatile constituents are either a mixture of monoterpene (limonene) and sesquiterpene, hydrocarbons and their oxygenated derivatives [24]. Traditionally, the fruits of *C. sinensis* serve as a rich source of vitamin C, thus potent in boosting the immune system with its rich stores of antioxidants [25]. Besides, they have been used in treating various ailments and conditions, including, constipation, cough, common cold, depression, and others [26]. Essential oils of *C. sinensis* have exhibited antibacterial, antifungal, anti-inflammatory, radical scavenging, and anxiolytic [27] effects.

### 3.2 Bitter orange (*Citrus aurantium* L.)

Bitter orange (*Citrus aurantium* L.) differs from sweet orange by the bitterness of its rind and the relatively high acidity of its pulp [22]. Its peel has been used as a source of flavor for soft drinks, baked foods, and liquors, and aromatizes a lot of drugs [28]. Different studies have reported D-limonene as the major volatile constituent of bitter orange oil, which forms about 93% of its composition, and other components including monoterpene, sesquiterpenes, oxygenated compounds, and aromatic alcohols [22, 29]. The concentrations of aliphatic aldehydes, oxygen-containing, monoterpenes, and sesquiterpenes in the ripe, bitter orange peel essential oil are much higher than in the unripe peels [30]. This species has been used in the treatment of several conditions such as anxiety, gastrointestinal disorders, and obesity [29, 31]. *C. aurantium* essential oils have shown significant biological properties, including radical scavenging, anticancer, anxiolytic, and antibacterial effects [5, 32].

### 3.3 Key lime (*Citrus aurantifolia* (Christm.) swingle)

*Citrus aurantifolia* is a flowering plant from the family Rutaceae, an important species in the genus *Citrus* commonly known as lime. It is a native of Southern Asia and commonly cultivated in the West Indies, the US, and South America [33]. The essential oil contains both terpenes and non-terpenoid derivatives and consists mainly of alcohol, esters, acids, aldehydes, amines, and oxides [34]. Chemical profiling investigations via steam distillation have shown that the peel oil contains nine (9) phytochemicals; Germacrene isomers (61.2%), Pinene (14%), Linalool dimer (2.9%), Bornane (11%), Citral (2.9%), Anethole (1.5%), Anisole (1.1%), Safole (0.3%), and Demitol (0.6%) [35]. These constituents and others are believed to contribute immensely to the medicinal properties of *C. aurantifolia* oil [36]. *C. aurantifolia* essential oils have promising biological properties such as antibacterial, anthelmintic, anti-inflammatory, anti-diabetic, and radical scavenging effects [5, 37].

### 3.4 Lemon (*Citrus limon* L. Osbeck)

It is a small evergreen medicinal plant that belongs to the genus *Citrus* and the family Rutaceae [38]. Lemon is commonly grown in cooler summer regions, with the USA and Italy being the largest producers. The main constituents of lemon essential oil are terpenes (78.8%); limonene (48%) and  $\beta$ -terpinene (17%) according to Sun, Chen, Zhang, Liu, Ma and Zhang [39]. The essential oil of *C. limon* consists of a mixture of alcohols, acids, aldehydes, and esters, and among the hydrocarbon monoterpenes, those that occur in appreciable quantities are D-limonene,  $\beta$ -pinene,  $\gamma$ -terpinene, and *p*-cymene [22]. GC/MS analyses of *C. limon* essential oil identified three phytochemical groups (monoterpene hydrocarbons (88.69%), oxygenated monoterpenes, and sesquiterpene hydrocarbons) [40]. In all, 37 volatile constituents were observed, with D-limonene (59.48%) and  $\beta$ -pinene (12.75%) as the major constituents. Traditionally, their essential oils have numerous health benefits and have been used as astringents, diuretics, antimigraines, and also for relief from fever, colds, and flu [41]. The lemon essential oil has been touted as a home health remedy as people claim can reduce anxiety and depression, ease morning sickness, promote healthier skin, act as a pain reliever, help breathe easier, soothe a sore throat, and help one feel alert and concentrated [42]. Most of these claims have been substantiated and confirmed in the literature. Various studies have reported the medicinal effects of *C. limon* essential oil which include antibacterial, antifungal, radical scavenging, and insecticidal [43, 44] activities.

### 3.5 Bergamot (*Citrus bergamia* Risso)

The *Citrus bergamia* Risso plant belongs to the genus *Citrus* of the family Rutaceae and known and used for centuries in south Italia (Calabria). It is reported to be a hybrid between lemon (*limon*, Burm.f.) and *Citrus aurantium* L. (Sour orange) or arises from a mutation of Lemon [45], and is well known for its unique fruits (bergamot). The essential oil from its peels is commonly used for perfume [6]. The greenish or reddish-brown colored essential volatile oil of bergamot is obtained by rasping and cold pressing and has a characteristic bitter taste and pleasant odor [46]. Bergamot essential is peculiar because of its phototoxic effect which is normally attributed to the presence of bergapten or 5-methoxy psoralen (5-MOP), a naturally occurring compound of the furanocoumarin (psoralens) family. Some of the major compounds in *C. bergamia* Risso essential oil include limonene, linalyl acetate, linalool,  $\gamma$ -terpinene, and  $\beta$ -pinene [47]. The oil also contains mainly monoterpene and sesquiterpene hydrocarbons, in addition to aliphatic aldehydes, alcohol, and esters [48]. Several studies have revealed the enormous biological potential of *C. bergamia* essential oil some of which include: anti-cholesterol, anxiolytic anticancer, anti-inflammatory, antifungal, and antibacterial effects [5, 49].

### 3.6 Grapefruit (*Citrus x paradisi* Macfady)

*Citrus paradisi*, also called grapefruit, is a citrus plant with bitter fruits commonly grown in the tropics and subtropics [50]. Reports showed that it was discovered in Barbados on the Island of the Caribbean in the early nineteenth century and was later brought to Texas in 1821 [51]. It has become a worldwide commercial citrus plant with more varieties being developed in some parts of the USA, specifically Florida and Texas [52]. Sabinene is the main constituent of grapefruit essential oils from the USA (Duncan and Marsh; 42–59%) [53]. Other constituents include ocimenes, linalool, terpinen-4-ol,  $\beta$ -pinene, limonene, and myrcene [22, 54]. Grapefruit essential oil has been ascribed to several medicinal properties, such as antibacterial, antifungal, anticancer, anti-obesity, radical scavenging, and insecticidal effects [5, 32] effects.

### 3.7 Mandarin Coleopatra *Citrus (Citrus reshni)*

This species of the citrus genus originates from India and was brought to Florida from Jamaica around the mid-nineteenth century [22]. The essential oil from the peels and fresh leaves obtained by hydrodistillation is yellow. The fruit has an acidic flavor and a red–orange color with a thin and rough texture of the peels [55]. The fruit peel has limonene as the major component (93.6%) and others like myrcene, sabinene, and linalool are also present in appreciable quantities. The leaves have sabinene (49.7%) as the main constituent, followed by linalool and beta ocimene [56]. *Citrus reshni* essential oil has been reported to exhibit anti-inflammatory, antimicrobial [57], and antiviral [58] activities.

### 3.8 Yuzu citrus (*Citrus junos Siebold ex Tanaka*)

*Citrus junos* is a small tree of the family Rutaceae that produces a yellow-golden colored fruit from which essential oil is obtained due to the sweet flavor. Yuzu is a Northeast Asian citrus plant that is believed to have originated from China and was brought to Japan in the tenth century [59, 60]. *C. junos* are grown in China, Korea, and Japan, and are mainly used in the food industry in the production of teas, wines, and juices [61]. The major compounds of yuzu essential oil are determined using gas chromatography–olfactometry, and recent studies revealed components of yuzu essential oil such as terpene hydrocarbons and their derivatives [62]. It has been used as traditional medicine in Japan for aromatherapy and also shows neuroprotective, anti-obesity, anticancer, anti-inflammatory, and radical scavenging [5, 32, 63] activities.

### 3.9 Kumquat citrus (*Citrus japonica*)

Kumquats (*C. Japonica*) is a citrus plant commonly grown in southern parts of China for its fruits [64]. This citrus plant produces edible fruit which is normally eaten together with the peels. It is characterized by a sweet aroma attributed to the presence of flavonoids and terpenoids [65]. The essential oils extracted by hydro-distillation are rich in flavonoids [64]. The volatile constituents of kumquat essential oil are sesquiterpenes, terpenes, alcohols, ketones, aldehydes, and esters, and the major component is limonene (93%) [65]. The volatile and semi-volatile compounds found in citrus essential oil represent 85–99% of the entire oil fraction, which accounts for about 200 [66]. Compounds most frequently reported in citrus essential oils are hydrocarbon, mono- and sesquiterpenes, aliphatic and olefinic C6–C12 non-terpene aldehydes, alcohols, ketones, esters, and acids, along with several aromatic compounds [66]. Modification of the chemical profile of the essential oil may be altered by the extraction method used, which could affect its biological properties. Some of its biological properties include anticancer, radical scavenging, and antibacterial and antifungal [5, 32] effects.

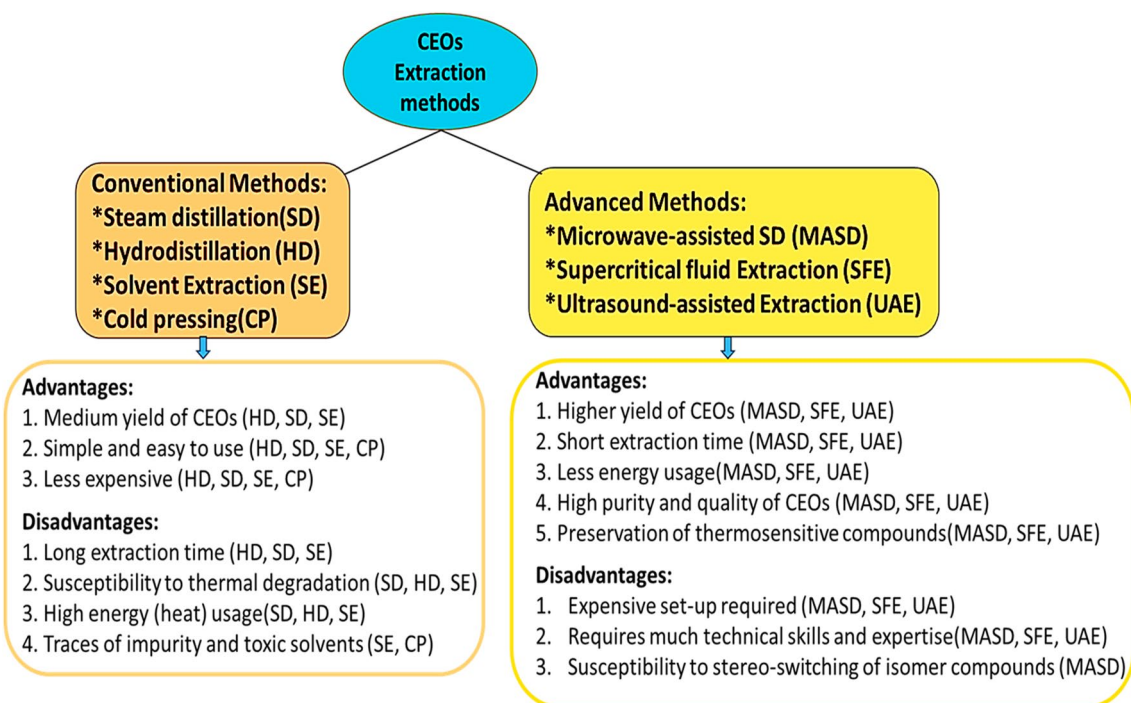
### 3.10 Mandarin orange (*Citrus reticulata Blanco*)

Mandarin (*Citrus reticulata Blanco*) citrus is a small fruit tree or shrub that grows up to an average height of 20 feet and it is believed to originate from India and China [67]. *C. reticulata* have glossy leaves with aromatic flowers and bear small light-yellow sweet-scented fruits that are easily detached [68, 69]. It is popularly referred to as ‘tangerine’ in most parts of the world and its fruit is tasty and eaten freshly. The fruits of *C. reticulata* have rich reserves of vitamins C, vitamin B1–B3, beta-carotene, folate, potassium, and magnesium minerals [70]. Aside from the fruits, the leaves, flowers, and seeds of the plant have been ascribed with medicinal properties such as anti-inflammatory, antioxidant, anticancer, anti-emetic, pain relieving, and laxative effects [71, 72]. The essential oil of *C. reticulata* has been reported to have antibacterial, antifungal, antiparasitic, and anti-cancer [32, 71] effects. The biological properties of *C. reticulata* have been attributed to the constituents of its essential oil such as limonene,  $\beta$ -pinene, myrcene, D-limonene, citropten, linalool,  $\alpha$ -thujene,  $\alpha$ -terpene, camphene,  $\alpha$ -pinene, 3-carene and tangerentin, and hesperidin [73, 74].

### 3.11 Overview of citrus essential oils extraction and detection methods

Citrus essential oils are normally synthesized as secondary metabolites stored within oil sacs found in peels, leaves, cuticles, and seeds [75]. These essential oils are extracted from the storage parts of the plant and purified using different extraction methods and techniques deemed efficient. The conventional methods of extraction for CEOs include solvent extraction, cold-pressing or hand pressing, simple distillation, steam distillation, and hydro-distillation

methods [2]. These conventional methods are mostly time-consuming and involve high temperatures and harmful solvents, which often lead to reduced yield, loss of volatile compounds, and reduced quality (Fig. 1) [76]. New and innovative extraction methods and techniques have gained attention in recent times due to the low cost of energy and reduced carbon dioxide emissions. Due to the shortcomings of the conventional methods of extracting CEOs, pharmaceutical, food, and chemical industries and research scientists are working assiduously to develop novel extraction techniques that are efficient, safe, sustainable, energy-efficient, and time-saving. These new methods will increase extraction efficiency by reducing the loss of volatile compounds and waste generation while requiring less energy (heat), and increasing yield and quality (Fig. 1) [2, 77]. Some of the new and efficient extraction methods include microwave-assisted steam distillation (MASD), Microwave hydro-diffusion and Gravity (MHG), ultrasound-assisted extraction (UAE), and supercritical fluid extraction methods [78]. These new methods are termed “green extraction methods” due to the reduced emission of carbon dioxide (CO<sub>2</sub>) and lower energy consumption, hence making them eco-friendly [79]. These newly developed methods and techniques are meant to achieve maximum extraction of the CEOs and their vital compounds in high quantities while reducing cost and pollution [77]. Currently, the MASD and UAE methods are reportedly the most efficient methods for the extraction of CEOs. After extraction of Citrus essential oils, the oil is subjected to a purification process to remove certain waste to increase the quality and enrich the concentration of targeted compounds. Chromatography methods are mostly used in the purification process. The major techniques are column chromatography, high-performance liquid chromatography (HPLC), and high-speed counter-current chromatography (HSCC) [80]. Also, some solvent combinations are used to enhance the purification processes, including chloroform and methanol, hexane and n-butanol or ethyl acetate, and butanol and water mixtures [77]. Purified CEOs containing the desired phytochemicals are detected and quantified using various methods such as mass spectroscopy (MS), gas chromatography-mass spectrometry (GC–MS), HPLC, nuclear magnetic resonance (NMR), UV-ultraviolet, ultra-high performance liquid chromatography time-of-flight mass spectrometry (UHPLC-ToF–MS), Field-emission scanning electron microscopy (FESEM), and attenuated total reflection Fourier-transform infrared spectroscopy (ATR-FTIR) (Table 1) [2, 77].



**Fig. 1** a figure showing the advantages and disadvantages of conventional and advanced methods of extraction of citrus essential oils (CEOs)

**Table 1** Ten selected citrus species and the methods of extraction and purification of their essential oils

Citrus species	Extraction method	Plant part	Purification/detection method	References
<i>C. aurantifolia</i> (lime)	Steam Distillation (SD)	Fruit, Epicarps, Flavedo, peels, leaves	GC, GC/MS	[35, 88–90, 99, 232, 233]
	Hydrodistillation (HD)/HD Cleverger apparatus	Peels, fruits, leaves, pomaces	GC/MS, UV-VIS, GC-FID	[37, 90, 106, 119, 121, 133, 150, 234–241]
	Hand/Mechanical Pressing	Pericarp	GC-MS	[168, 242]
	Microwave-assisted Steam Distillation (MASD)	Peels, leaves	GC-MS	[106, 243]
	Solvent/Maceration extraction	Peels	FTIR, GC-O, GC-MS	[183, 192]
	Microwave hydro-diffusion and Gravity (MHG)	Fruits peels	GC, GC-MS	[161]
	Supercritical fluid extraction, SC-CO <sub>2</sub>	Leaves	GC-MS, FTIR, UV	[207]
	Ultrasound-assisted Extraction (UAE)	Fruit peels	HPLC	[227]
	Steam distillation (SD)	Fruits, peels, blossoms	GC-FID, GC-MS, HS-GC-MS	[94, 95, 228, 244–247]
	Hydrodistillation (HD)/HD Cleverger apparatus	Peel, Flower, leaves, blossoms	GC-MS, GC-FID, GC-MS-LRI, CPG-MS	[129, 152, 230, 240, 241, 246–251]
<i>C. aurantium</i> (Bitter orange)	Microwave-assisted hydrodistillation (MA-HD)	Peels	CGP-MS	[152]
	Microwave-assisted steam distillation (MASD)	Peels	GC-MS	[94, 252]
	Ultrasound-assisted Extraction (UAE)	Buds	GC-MS	[228]
	ultrasonic-assisted headspace solid phase micro-extraction (UA-HS-SPME)	Flower	GC-MS	[230]
	Cold pressing	Fruit cortex	GC-MS	[164, 168]
	Solvent-free microwave-assisted extraction (SFME)	Blossoms	GC-FID, GC-MS	[247]
	Ohmic-assisted hydrodistillation (OAH)	Blossoms	GC-FID, GC-MS	[247]
	Supercritical CO <sub>2</sub> (SC-CO <sub>2</sub> )	Peels	GC-FID/MS	[206]

Table 1 (continued)

Citrus species	Extraction method	Plant part	Purification/detection method	References
<i>C. bergamia</i> (bergamot)	Steam Distillation (SD)	Fruits	GC-MS	[96]
	Hydrodistillation (HD)/HD-Clevenger apparatus	Peels	GC-FID, GC-MS	[130, 241, 253]
	Headspace solid-phase microextraction (HS-SPME)	Peels	GC-MS	[231]
	Supercritical CO <sub>2</sub> (SC-CO <sub>2</sub> )	Peels	GC-FID/MS, GC-MS	[48, 206]
	Supercritical fluid extraction (SFE)	Seeds	GC	[208]
	Solvent/Soxhlet Extraction	Seed, peels	GC, LC-MS	[208, 254, 255]
	Hand/Cold pressing	Peels, fruits	GC, GC-MS, LCMS-IT-ToF, GC-FID, GC-C-IRMS, enantio-GC, MDGC, HPLC and HPLC-MS-IT-ToF	[165, 168, 240, 256–259]
	ultrasound-assisted hydrodistillation (UAHD)	Peels	GC-MS	[231]
	Steam Distillation (SD)	Peels	GC, GC-FID, GC-MS	[86, 90–92, 99, 101, 107, 194]
	Microwave-assisted steam distillation (MASD)	Peels	GC-FID, GC-MS	[86, 194, 252]
<i>C. sinensis</i> (Sweet orange)	Microwave hydro-diffusion and Gravity (MHG)	Peels	GC-MS	[161]
	Hydrodistillation (HD)/HD Clevenger apparatus	Peels	GC, GC-MS HPLC, FTIR, GC-FID FESEM, CPG-MS	[92, 107, 122–128, 134, 144, 146, 152, 224, 241, 251, 260, 261]
	Cold pressing	Peels	GC-FID, GC, GC-MS	[122, 163, 165, 166, 168, 169, 176, 259, 262]
	Microwave-assisted hydrodistillation (MA-HD)	Peels	GC, GC-MS, CPG-MS, DRIFT	[127, 152, 153, 196, 197]
	Salt-assisted extraction hydrodistillation (S-HD)	Peels	GC-MS	[156]
	Enzymes-assisted hydrodistillation (E-HD)	Peels	GC-MS, GC	[155, 156]
	Solvent free Microwave extraction (SFME)	Peels	GC-MS, GC-FID, DRIFT	[122, 123, 156, 201]
	Microwave-assisted solvent extraction (MASE)	Peels	GC-MS	[200]
	Ionic liquid-based microwave-assisted extraction (MAE-IL)	Peels	GC-MS	[199]
	Solvent/Soxhlet Extraction	Peels	GC, GC-MS, FTIR	[92, 185–187, 260]
Ultrasound-assisted extraction (UAE)	Peels, pulp	GC, GC-MS, HPLC, FTIR	[156, 224, 260, 263]	
Supercritical CO <sub>2</sub> (SC-CO <sub>2</sub> )	Peels	GC-FID/MS	[206]	

Table 1 (continued)

Citrus species	Extraction method	Plant part	Purification/detection method	References
<i>C. limon</i> (Lemon)	Steam distillation (SD)/SD Cleverger apparatus	Peels, rind, leaves, fruits, epicarps	GC, GC-FID, GC-MS	[86, 90, 93, 98–103, 232, 244, 264]
	Microwave-assisted steam distillation (MASD)	Peels	GC-FID, GC-MS	[86, 252]
	Cold/Hand pressing	Peels, seeds, leaf	GC, GC-MS, GC-FID, RP-HPLC, GC-C-IRMS	[165, 167–171, 252, 259]
	Solvent extraction/S Soxhlet Extraction	Peels, leaves	GC-MS	[136, 188, 189]
	Microwave hydro-diffusion and Gravity (MHG)	Peels	GC-MS	[161]
	Hydrodistillation (HD)/HD Cleverger apparatus	Peels, zest, leaves, aerial parts	GC, GC-MS, CPG-MS, GC-FID, FTIR, DRIFT	[120, 128, 131, 132, 134–138, 140–145, 152, 158, 189, 195, 239, 241, 251, 252, 265, 266]
	microwave-assisted hydrodistillation (MAHD)	Fruit peels	GC-MS, CPG-MS	[120, 152, 153, 158, 195]
	Plasma and microwave-assisted hydrodistillation (PAHD)	Peels	GC-MS	[158]
	Ohmic heating-assisted hydrodistillation (OH-HD)	Peels	GC, FTIR	[151]
	Solvent-free microwave extraction (SFME)	Peels, leaves	GC-MS, DRIFT	[120, 195, 201, 265]
<i>C. junos</i> (Yuzu)	Supercritical fluid extraction (SFE)/SC-CO <sub>2</sub>	Peels and leaves	GC-MS, GC-FID	[136, 145]
	Simultaneous distillation extraction at reduced pressure (V-SDE) and Solid-phase microextraction	Peels	MDGC-MS	[267]
	Soxhlet/ Solvent extraction	Peels, pomace	GC-MS	[190]
	Hexane Soxhlet with Supercritical CO <sub>2</sub> extraction	Seeds	GC-MS, GC-FID	[209]
	Supercritical CO <sub>2</sub> (SC-CO <sub>2</sub> )	Peels	GC-MS, GC-FID	[59, 210, 211]
	Cold pressing	Peels	GC-MS	[174, 175, 268]

Table 1 (continued)

Citrus species	Extraction method	Plant part	Purification/detection method	References
<i>C. paradisi</i> (Grapefruit)	Steam distillation (SD)	Peels	GC-MS	[101]
	Solvent/Soxhlet extraction	Peels	GC, GC-MS	[186, 269]
	Microwave-assisted steam distillation (MASD)	Peels	GC-MS	[252]
	Hydrodistillation (HD)/HD Clevenger apparatus	Peels, Zest, rind	GC-MS, FTIR	[54, 134, 139, 142, 146, 147, 157, 202, 241, 261, 270, 271]
	Enzyme-assisted hydrodistillation (E-HD)	Peels	GC	[155]
	Microwave-assisted hydrodistillation (MAHD)	Peels	GC-FID/GC-MS	[157]
<i>C. reticulata</i> (Mandarin)	Microwave hydro-diffusion and gravity (MHG)	Peels	GC-MS	[161]
	Cold/Hand pressing	Peels	GC, GC-MS	[139, 165, 169, 172]
	Solvent-free microwave extraction (SFME)	Peels	DRIFT, GC-MS	[201, 202]
	Steam distillation (SD)/SD Clevenger apparatus	Fruits, rinds, peels	GC-FID, GC-MS	[86, 101, 103, 244]
	Hydrodistillation (HD)/HD Clevenger apparatus	Peel, leaf, Zest	GC-MS, NMR, GC-FID, HPTLC-densitometry	[56, 142, 146, 149, 241, 261, 272–275]
	Hand/mechanical pressing	Peels	GC, GC-MS	[72, 165, 168]
	Microwave-assisted steam distillation (MASD)	Peels	GC-FID	[86]
	Microwave-assisted hydrodistillation (MAHD)	Peels	GC-MS	[67]
	Ultrasound-assisted extraction (UAE)	Fruit peels	HPLC, FTIR	[227, 276, 277]
	Hydrodistillation (HD)/HD-Clevenger apparatus	Peels, leaves	GC-MS, GC-FID, NMR	[64, 154, 191, 198, 229, 251, 253, 278–281]
<i>Fortunella crassifolia</i>	Steam Distillation	Fruits	GC-MS, HPLC	[104]
	Microwave-assisted hydrodistillation (MAHD)	Leaves	GC-MS, C NMR	[154]
<i>C. japonica</i>	Solvent extraction	Peels	GC, GC-MS	[191, 282]

Table 1 (continued)

Citrus species	Extraction method	Plant part	Purification/detection method	References
<i>F. Margarita</i>	Vacuum hydrodistillation	Peels	GC-MS	[229]
	Cold pressing	Peels	HS-SPME/GC	[105]
	Hydrodistillation (HD) / Clevenger apparatus	Peels, leaves	GC-MS	[283, 284]
	Steam distillation	Peels	HS-SPME/GC	[105]
	Ultrasound-assisted vacuum hydro-distillation (UAV-HD)	Peels	GC-MS	[229]
	Ultrasound-assisted hydrodistillation (UA-HD)	Peels	GC-MS	[198]
	Microwave-assisted hydrodistillation (MA-HD)	Peels	GC-MS	[198]
	Supercritical CO <sub>2</sub> extraction	Peels	GC-MS	[191, 212]

## 4 Methods of extraction of *citrus* essential oils (CEOs)

### 4.1 Steam distillation (SD)

This is one of the most common methods used in the large-scale production of various essential oils and about 90% of essential oils are extracted with this technique [81]. This method is a modification of a normal distillation system where a generated steam interacts with the plant material to release the targeted substance, and it is suitable for extracting heat-sensitive compounds and substances [82]. It involves the use of a steam generator flask, a distilling flask, a condenser, and a receiving vessel. This method has been employed officially for extracting various citrus essential oils because of its high efficiency and is also economical compared to other methods [83, 84]. The method involves feeding the plant materials, mostly leaves or fruit peels into the chamber (a perforated plate) above the steam inlet without exposing the plant material to a direct heat source but rather to vapor from the boiler [85]. This is followed by condensation in a condenser which separates the oily molecules from water vapor [86]. Steam distillation was applied in the extraction of essential oils from the chopped leaves or peels of lemon, mandarin, and orange by passing a steam (1 kg/h; 96–100 °C) at 1.1 atm for about 3 h [87]. Steam distillation was slightly modified by preheating before the distillation and it gave a higher yield of essential oil from *C. sinensis* and *C. reticulata* at a percentage of 4.24% and 5.87% respectively, as compared to the conventional steam distillation which yielded 2.48% and 0.98% for *C. sinensis* and *C. reticulata* respectively [83]. The steam distillation method has also been used to extract essential oil from the peels, flavedo, and leaves of *C. aurantifolia*, *C. limon*, *C. aurantium*, *C. bergamia*, *C. limetta*, *C. reticulata*, *F. crassifolia*, *F. margarita*, *C. sinensis* and *C. aurantifolia* (Table 1) [86, 88–106]. A steam explosion method operating at high temperature and pressure was used to extract essential oil from the fruit peels of *C. sinensis* and was found to reduce extraction time by eightfold in comparison to a hydrodistillation method [107]. Even though this method gives a substantial yield of safe and quality essential oil from various citrus plant materials. The major setbacks of the steam distillation method are the vapour pressure and the thermal degradation of thermolabile compounds, longer extraction time, and high energy consumption, which have also been reported to induce partial hydroxylation [108, 109]. The disadvantages associated with this method have led to newly improved “green” extraction techniques such as ultrasound-assisted extraction (UAE), which was faster with increased quality compared to the conventional method [110]. Others like microwave-assisted extraction (MAE) and CO<sub>2</sub> supercritical extraction have also been more efficient than the old extraction methods [111, 112].

### 4.2 Hydrodistillation (HD)

This method is the most common method employed in the extraction of essential oil [113]. In this method, the plant material is placed in the water bath and boiled to the boiling point (mostly 100 °C) under atmospheric pressure. The mixture is then condensed by cooling, followed by separation of the mixture via decantation [114]. This method is efficient for plant materials with water-soluble and thermostable constituents at high boiling points, but very high temperatures could degrade heat-sensitive components, thus affecting the quality of the essential oil [115, 116]. HD is reported to be more efficient than other methods because it does not involve the use of chemical solvents, and thus the toxic effect of chemicals and the subsequent loss of volatile compounds during the removal of solvents is easily avoided [117]. In other studies, *C. aurantium*, *C. japonica*, *C. sinensis*, *C. aurantifolia*, grapefruit (*C. paradisi*), *F. margarita*, *C. reticulata*, kumquat (*F. margarita*), *C. bergamia* and lemon (*C. limon*) fruit peels and leaves and essential oils were extracted using hydrodistillation (Table 1) [74, 90, 92, 118–149]. In another study, *C. limon* peels and distilled water were placed in the HD with a Clevenger-type apparatus, and it was followed by condensation which separated the oily molecules from that of water vapour [120]. In a similar procedure, the fruit peels of *C. aurantifolia* and *C. limon* gave an average yield of 1.4% from 150 g of fruit peels [150]. The oven-dried peels of *C. reticulata*, *C. sinensis*, and *C. paradisi* yielded substantial quantities in the following ranges from 0.30–0.50 g/100 g, 0.24–1.07 g/100 g and 0.20–0.40 g/100 g, respectively, under the hydrodistillation method [118]. In a comparative study, the hydrodistillation method used for *C. sinensis* essential oil extraction produced oil that contained 10 to 13 main constituents which were absent in the steam explosion method. However, the steam explosion method was eight times faster than the hydrodistillation method [107].

The Ohmic heating-assisted hydrodistillation method was successfully used to co-extract essential oil and pectin from *C. limon* waste peels [151]. Several modifications of the traditional hydrodistillation method have led to

decreased extraction time and enhanced extraction efficiency. These include turbo-hydro-distillation, ultrasound-assisted hydrodistillation (U-HD), enzyme-hydrodistillation (E-HD), micelle-hydrodistillation, and salt-hydrodistillation (S-HD). In a recent study, the microwave-assisted hydrodistillation (MA-HD) method was used to extract essential oils from the fruit peels of *C. aurantium*, *F. japonica*, *C. limon*, and *C. sinensis*, and the yield was almost twice the yield in the conventional hydrodistillation method [152–154]. Interestingly, another study reported that the enzyme-assisted hydrodistillation technique enhanced essential oil recovery yield from *C. paradisi*, *C. sinensis*, and *C. limonium* cv. colima and the extraction efficiency was between twofold to sixfold that of the conventional hydrodistillation method [155]. The microwave-assisted hydrodistillation (MAHD), ultrasound-assisted hydrodistillation (U-HD), enzyme-assisted hydrodistillation (E-HD), and salt-assisted hydrodistillation (S-HD) methods were used to extract essential oil from the peels of *C. sinensis* [127, 156]. In a comparative study, the fruit peels essential oil of *C. paradisi* was extracted through hydrodistillation (HD) and microwave-assisted hydrodistillation (MAHD) and the GC-FID/GC-MS analysis shows that MAHD possessed 28 major constituents as against 21 constituents in the HD [157]. Hydrodistillation (HD), microwave-assisted hydrodistillation (MAHD), and plasma and microwave-assisted hydrodistillation (PAHD) methods were used comparatively to extract essential oil from the peels of *C. limon*. The results from the study revealed that the plasma-assisted microwave pre-treatment (PAHD) and microwave-assisted hydrodistillation (MAHD) methods significantly improved the extraction yield by 149.34% and 58.28% as compared to hydrodistillation (HD) [158].

### 4.3 Cold pressing

Hand or cold pressing, also known as mechanical pressing, is one of the oldest traditional methods of extracting citrus essential oils in large quantities before the emergence of the use of scientific apparatus, methods, and techniques. The essential oil obtained by cold pressing is characterized by strong aroma properties, high nutritional value, and high levels of major constituents because it is free from heat and chemical treatment [159]. The cold pressing method is done manually by pressing the peel layers exposing the oil sacs. Followed by squeezing the oil out of the sacs into the form of a watery emulsion and it is centrifuged to obtain the separate essential oil [85, 160, 161]. In this method, citrus essential oils could also be squeezed from the fruit peels using machines at room temperature and subsequently, washed with cold water. Finally, the oil is then obtained by centrifugation [84]. Although mechanical or hand pressing is one of the traditional methods of extracting essential oils, it is not a preferred method due to the low yield compared to the steam and hydrodistillation methods [85]. However, cold pressing was employed in extracting essential oils from *C. aurantium*, *F. margarita*, *C. sinensis*, *C. junos*, *C. reticulata*, *C. limon*, *C. paradisi*, and *C. grandis* by pressing the follicular glands of citrus peels through mechanical maceration thus releasing their contents (Table 1) [23, 59, 105, 122, 139, 162–176]. This method is reported to be simple, rapid, environmentally friendly, and inexpensive compared to alternative methods [160]. Cold-pressed oils, aside from being safe, also have a characteristic aromatic fragrance which is the reason behind their wide application in the beverages, cosmetics, and pharmaceutical industries [177]. However, the cold pressing method is not suitable for plant materials with low essential oil content. Cold-pressed oils may also contain polyunsaturated fatty acids (PUFAs) and pro-oxidative components that reduce their oxidative stability and shelf-life [178].

### 4.4 Solvent extraction (SE)

This is the oldest method used to obtain a crude extract from plants and also has been used in essential oil extraction via the principle of solubilization of compounds present in the cells of various plant materials using an appropriate solvent [179]. Essential oils extracted under this method normally involve dissolving the plant material in a specific solvent and the oil is obtained by subjecting it to evaporation followed by filtration and distillation [114]. Solvents like chloroform, ethanol, acetone-hexane, and ethyl acetate have been used in the extraction of various essential oils including citrus essential oils [83, 180, 181]. Thus, the fruit peels of *C. reticulata* were successively extracted with hexane, chloroform, and acetone using a Soxhlet extractor [180]. In another study, ethyl acetate was employed in extracting CEOs [182]. Citrus essential oil was extracted from fruit peels of lime (*C. aurantifolia*) using ethanol, hexane, and water in a maceration solvent and Soxhlet extraction methods [183]. Also, the solvent extraction method gave a substantial yield of essential oil from the peels of *C. medica* compared to the hydrodistillation method [184]. In addition, the essential oil was extracted from fruit peels and leaves of *C. sinensis*, *C. junos*, *F. margarita*, *C. limon*, and *C. paradisi* by Soxhlet solvent extraction method using hexane, n-hexane, and ethanol (Table 1) [92, 136, 185–191]. *Citrus aurantifolia* Swingle fruit peels were extracted with pentane: ether (1:1) solvent extraction method and over 50 volatile compounds were identified using

GC-O and GC-MS [192]. Hexane was used to extract essential oil from the fruits of *Citrus limetta* which gave a higher yield than steam distillation. However, the oil obtained contained pigments, waxes, and polysaccharides, hence reducing its quality [97]. The main problem associated with this method is the toxic effects of some of the solvents such as benzene and dichloromethane commonly used, coupled with the loss of certain volatile compounds during the removal of the solvents [193]. Thus, the method also requires heating the solvent to 100°C and however, not suitable for extracting volatile and thermolabile components [114].

#### 4.5 Microwave-assisted steam distillation (MASD)

This method is a modified form of steam distillation method that has been used in some studies to increase the yield extraction of CEOs and preserve specific components that are susceptible to destruction by direct steam. The main principle of this method relied on the switch in polarity from water-generated steam to wave-generated steam, where the normal heating source (electric heating cap) has been replaced with a microwave [179]. The MASD works through fast rupture of the cell wall, thus releasing the content of the cytoplasm using strong microwaves [4]. The method is said to be environmentally friendly for the extraction of essential oils from different plant materials [111]. Essential oils from orange (*C. sinensis*), lemon (*C. limon*), mandarin (*C. reticulata*) obtained by MASD yielded 1.150%, 1.115%, and 0.940% respectively [86]. This method has been used to extract essential oil from the peels of *C. limon*, *C. reticulata*, *C. sinensis* fruits [86, 194]. Also, the microwave-assisted hydrodistillation method was successfully used to extract essential oil from the fruit peels of *C. reticulata* and the GC-MS analysis revealed that the oil contained limonene,  $\beta$ -myrcene, and sabinene in a proportion of 97.68%, 1.39% and 0.12% respectively [67]. Another study reported that MASD has a shorter extraction time (about 6 min) and yields much higher quality essential oil with strong aromatic properties from *C. sinensis* peels at a microwave power of 500W compared to the traditional steam distillation method [194]. The solvent-free microwave extraction method was employed in the essential of essential oil from the fruit peels of *C. aurantium*, *C. sinensis*, and *C. limon* (Table 1) [94, 156, 195]. Microwave-assisted steam distillation has higher efficiency than steam distillation because it shortens extraction time and enhances the process of extraction while preserving the components of the oil in higher quantities [4]. The shortcoming reported in this method had to do with the use of microwaves since the waves are believed to result in chemical stereo-switching from one isomer to another in the compounds found in the essential oils, thus altering certain key components to other forms [179]. Besides MASD, other microwave-assisted extraction methods used in the extraction of *C. sinensis* essential oils include hydro-diffusion and gravity (MHG) [161]. The microwave-assisted hydrodistillation (MA-HD) method was employed in the extraction of essential oils from *C. limon*, *F. margarita*, *C. sinensis*, and *C. medica* [120, 184, 195–198]. Ionic liquid-based microwave-assisted extraction (MAE-IL), solvent-free microwave extraction (SFME), and microwave-assisted solvent extraction (MASE) were successfully used to extract essential oils from the peels of *Citrus sinensis* [122, 123, 156, 199, 200]. Solvent-free microwave-assisted extraction (SFME) has been employed in extracting essential oil from the peels of *C. limon*, *C. sinensis*, and *C. paradisi* and the yield was higher than that of the industrial extraction methods [201, 202]. Methods like ultrasonic microwave-assisted extraction, vacuum microwave-assisted extraction, and microwave-assisted Soxhlet extraction have not been used to extract citrus essential oils (CEOs) yet.

#### 4.6 Supercritical fluid extraction (SFE)

This is one of the complex methods of extracting essential oil from plant materials commonly used in food, cosmetics, and pharmaceuticals [109, 203]. The method operates based on using harmless gases, for example, carbon dioxide (CO<sub>2</sub>), propane, butane, and others at their supercritical stage (at high pressure) to extract essential oils [179]. These non-toxic solvents, including carbon dioxide (CO<sub>2</sub>), propane, butane, and others are used to extract essential oil from plant material at low temperatures and high pressure, hence, avoiding thermal destruction or breakdown of volatile constituents [204]. Carbon dioxide (CO<sub>2</sub>) solvent is commonly used in the supercritical fluid extraction (supercritical carbon dioxide, SC-CO<sub>2</sub>) of citrus essential oil because of its characteristic low surface tension, high diffusivity, viscosity, and low critical temperature (304 K) [205]. Moreover, the method is termed a 'green' technology application and has been efficiently used to extract some CEOs, specifically, SC-CO<sub>2</sub> was used to obtain quality essential oils from, *C. limon*, *C. aurantifolia*, *C. bergamia*, *C. aurantium*, *F. margarita*, *C. sinensis*, *C. sphaerocarpa*, and *C. junos* essential oils (Table 1) [109, 136, 145, 191, 205–212]. The SFE method is superior to other methods because it is toxic-free, nonflammable, and requires low to moderate temperature and pressure [213]. However, the main problem associated with the use of SC-CO<sub>2</sub> is that it has a low polarity which affects the extraction of polar compounds (analytes).

Hence, certain modifications have to be made through the addition of polar modifying solvents like methanol or ethanol to enhance solution power and subsequently improve extraction efficiency [203]. Moreover, the SC-CO<sub>2</sub> extraction process involves the pretreatment of plant material that includes milling and drying, thus, exposing the plant material to air oxidation and enzymatic denaturation [109]. Furthermore, this method is very expensive and has slow extraction kinetics due to the difficulty of using mechanical vibrations [214].

#### 4.7 Ultrasound-assisted extraction (UAE)

The use of ultrasound waves for the extraction of phytochemicals has gained attention in recent times due to its efficiency and eco-friendly nature. The ultrasound-assisted extraction (UAE) method has been successfully used for lipids, proteins, and bioactive compounds such as polyphenols, berberine, and citrus essential oils [215, 216]. In the UAE extraction, the plant material such as the leaves, flowers, fruit peels, and seeds are soaked in water and other solvents like ethanol, and the ultrasound is then applied [217]. The UAE works on the principle of acoustic cavitation which involves the production, expansion, and rupturing of bubbles filled with gaseous vapor in liquid which facilitates the disruption and permeation of the cell wall of the plant tissue or extraction material [218, 219]. This cavitation process induces turbulence and current circulation in the liquid, resulting in the compression and refraction of the gas-filled bubbles, thus enhancing the rate of extraction at ambient temperature and pressure [220, 221]. This green extraction method has become one of the choicest methods in most industries because of its associated benefits, which include reduced energy consumption, heat production, solvent usage, waste generation, processing time, and increased yield and quality [222, 223]. This clean extraction method has been applied in the extraction of citrus essential oils, including *C. sinensis*, *C. limetta* (sweet lime), *C. aurantium*, *F. margarita*, *C. aurantifolia*, and *C. reticulata* (Table 1) [3, 198, 216, 224–229]. Ultrasound and microwave methods have been used simultaneously as one method referred to as the ultrasonic microwave extraction method to extract essential oils. Ultrasonic microwave-assisted hydrodistillation extraction (UMHD) was used to extract essential oil from the peels of *Citrus medica* and *Fortunella margarita* [184]. The study revealed that the UMHD method and the hydrodistillation method had similar essential oil composition, however, the UMHD increased the extraction yield and reduced extraction time. Ultrasonic-assisted headspace solid phase micro-extraction (UA-HS-SPME) has been used to extract essential oil from *C. aurantium* and *C. bergamia* [230, 231]. Another study used ultrasound-assisted hydrodistillation (UAHD) to extract essential oil from the fruits of *Citrus bergamia* which gave an extraction yield of 0.48%, which was estimated to be 118% higher than the yield from hydrodistillation (HD) [231].

## 5 Conclusion

Over the years there has been a significant improvement in techniques employed in the extraction of citrus essential oils (CEOs). The traditional extraction methods are associated with limitations such as long extraction time, high energy consumption, CO<sub>2</sub> emissions, degradation of major essential oil constituents, and reduction in oil yield and quality. However, technological advancement has helped CEO extraction through the development of innovative, efficient, and eco-friendly extraction techniques. As new methods continue to evolve each day, techniques such as ultrasound, microwaves, and supercritical fluid extraction have demonstrated promising extraction efficiency and could be used for large-scale production of quality CEOs. This present review gives an in-depth exposition of the extraction techniques, their advantages, and disadvantages, as well as the innovations and modifications of the current techniques developed for the extraction of citrus essential oils (CEOs). This review, therefore, brings to the forefront the most recent efficient extraction techniques and modifications for CEOs and provides a well-organized and informative extraction choice for researchers and industries.

**Authors contributions** AB and CKA wrote the main manuscript text and CO and CKA reviewed the manuscript.

**Funding** This research did not receive funding from any public or non-profit agencies.

**Data availability** No datasets were generated or analysed during the current study.

## Declarations

**Competing interests** The authors declare no competing interests.

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