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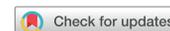
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## Extraction and purification of $\alpha$ -pinene; a comprehensive review

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### ABSTRACT

Extensive use of  $\alpha$ -pinene in cosmetics, and medicine, especially for its antioxidant/antibacterial, and anti-cancer properties, and also as a flavoring agent, has made it a versatile product.  $\alpha$ -Pinene (one of the two pinene isomers) is the most abundant terpene in nature. When extracting  $\alpha$ -pinene from plants and, to a lesser extent, fruits, given that its purity is essential, purification methods should also be used as described in this study. Also, an attempt has been made to describe the extraction techniques of  $\alpha$ -pinene, carried out by conventional and novel methods. Some disadvantages of conventional methods (such as hydrodistillation or solvent extraction) are being time consuming, low capacity per batch and being labor intensive and the requirement of trained operators. Most novel methods, such as supercritical fluid extraction and microwave-assisted extraction, can reduce the extraction time, cost, and energy compared to conventional methods, and, in fact, the extraction and preservation efficiency of  $\alpha$ -pinene in these methods is higher than conventional methods. Although the above-mentioned extraction methods are effective, they still require rather long extraction times. In fact, advanced methods such as green and solvent-free ultrasonic-microwave-assisted extraction are much more efficient than microwave-assisted extraction and ultrasound-assisted extraction because the extraction efficiency and separation of  $\alpha$ -pinene in these methods are higher; furthermore, no solvent consumption and maximum extraction efficiency are some crucial advantages of these techniques. However, the application of some novel methods, such as ultrasound-assisted extraction, in industry scale is still problematic because of their intricate design data.

### KEYWORDS

Conventional techniques;  
extraction; novel methods;  
 $\alpha$ -Pinene; purification; terpens

### Introduction

The search for essential oils (EOs) of aromatic plants worldwide is growing due to their potential applications in the food, health, cosmetic, and pharmaceutical industries (Dhifi et al. 2016; El Khetabi et al. 2022; Moura et al. 2022; Ramsey et al. 2020; Sankarikutty and Narayanan 2003). In recent years, the popularity of EOs has increased due to their antimicrobial, antiviral, anti-inflammatory, antibiotic, and antioxidant properties, as well as some favorable effects such as reducing stress, treating depression, and helping with insomnia (Dhifi et al. 2016). EOs, composed of volatile organic compounds (VOCs), belong to various chemical classes such as alcohols, aldehydes, ketones, ethers, amines, amides, heterocycles, phenols, esters, and mainly terpenes (Dhifi et al. 2016). One of the most essential components in EOs is terpenes. Monoterpenes are significant fragrant molecules widely distributed in nature, which can be isolated from the flowers, leaves, and fruits of plants (Alcántara, Hernaiz, and Sinisterra 2011). Experimental

studies have shown that some monoterpenes have anti-carcinogenic properties and act at different cellular and molecular levels. They can also be potentially considered as effective and nontoxic agents of anti-tumor diet (despite not being applied in clinical practice yet), which is promising as a new class of anti-cancer drugs (Loza-Tavera 1999).

Pinene (C<sub>10</sub>H<sub>16</sub>), either  $\alpha$ - or  $\beta$ -pinene, is a bicyclic, double-bond, terpenoid hydrocarbon (Winnacker 2018). It is used as a flavoring agent, insect repellents and, also in cosmetics and perfumery (Chen, Vermaak, and Viljoen 2013; Risner et al. 2020; Yang et al. 2013).  $\alpha$ -Pinene could be produced from several ways; indeed, according to recent studies, in addition to fruits and vegetables,  $\alpha$ -pinene can be produced from whey permeate (Risner et al. 2020).  $\alpha$ -Pinene used in industry is mainly obtained by tapping trees (gum turpentine) or byproduct of paper pulping (Yang et al. 2013). A common strategy for the microbial generation of pinene is the transformation of plasmids containing genes isolated from plants (Niu et al. 2018; Sarria et al. 2014; Vickers et al. 2015).

Therefore, according to this article, it can be said that extracting low levels of  $\alpha$ -pinene produced in plants by different methods can be very effective.  $\alpha$ -Pinene is produced industrially by fractional distillation of turpentine. (+)- $\alpha$ -Pinene is present in *Pinus palustris* Mill. and *Pinus caribaea* oils at concentrations of 65% and 70%, respectively (Noma and Asakawa 2010; Siddiqui et al. 2022).  $\alpha$ -Pinene in plants and fruits is extracted to a lesser extent with other compounds.

Some factors such as harvest time, harvest season, and type of drying affect the amount of  $\alpha$ -pinene. According to the obtained results in the literature, for the same population,  $\alpha$ -pinene continuously decreased from 25.7% at 6:00 am to 11.7% at 3:00 pm in the day (Pinho-da-Silva et al. 2010). Indeed, harvest time affects the yield and the use of  $\alpha$ -pinene. In addition, yield and utilization rates were higher in autumn harvest. Drying in the shade led to higher yields of  $\alpha$ -pinene (Matin et al. 2021). Furthermore, geographical areas (Central and South America, Mediterranean Europe and the Middle East, or South Africa) and plant type are among the factors that have been considered in the  $\alpha$ -pinene extraction method (Ascrizzi et al. 2017).

This article focuses on the conventional and novel extraction methods of  $\alpha$ -pinene and the advantages of each method as much as possible. Then, it compares different extraction ways of  $\alpha$ -pinene with each other. Also, this review gives a detailed description of varied techniques for the analysis of  $\alpha$ -pinene. Finally, it pinpoints various purification techniques of  $\alpha$ -pinene.

### $\alpha$ -Pinene; chemical structure, biological properties, and analysis

$\alpha$ -Pinene is one of the two isomers of pinene. It is found abundantly in the EOs of trees such as pine and is one of the most well-known compounds in the monoterpene family. It has two enantiomers (+) and (-), which are shown in Figure 1 (Salehi et al. 2019).  $\alpha$ -Pinene is a colorless liquid that is soluble in oil and ethanol but insoluble in water. It has been identified in at least 40 EOs and has a boiling point of 155°C (Berger 2007; Vespermann et al. 2017). Pinene is also used as a precursor for fuel production (George et al. 2015; Rivas da Silva et al. 2012; Wu and Maravelias 2018).

$\alpha$ -Pinene can be found in oils of coniferous trees, mainly pine (Vaz et al. 2016). This monoterpene is present in many plants such as lemongrass, mandarin, mint (*Satureja*

*cuneifolia*) and mintb (*Satureja montana*), Plai-Dam (*Zingiber ottensii*), rosemary, sage, tangerine, *Thymus longicaulis* subsp. *longicaulis* var. *longicaulis* (Tongnuanchan and Benjakul 2014), *Artemisia capillaris* Thunb, *Pinus koraiensis* Siebold & Zucc. (Pinaceae) (Sharifi-Rad et al. 2017).  $\alpha$ -Pinene has several properties, including antimicrobial, apoptotic, anti-metastatic, and antibiotic properties (Hernandez-Ledesma and Martinez-Villaluenga 2021). It could potentially be used to treat various inflammatory diseases (Kim et al. 2015). Furthermore, the inflammation associated with acute pancreatitis is reduced by  $\alpha$ -pinene in vivo via the downregulation of IL-6, IL-1 $\beta$ , and TNF- $\alpha$  (de Cássia da Silveira e Sá, Andrade, and de Sousa 2013; Hernandez-Ledesma and Martinez-Villaluenga 2021). Moreover, it has inhibitory effects on leukemia and breast cancer (Zhou et al. 2004). The antioxidant activity and cytotoxicity properties of  $\alpha$ -pinene have also been reported (Bouzenza et al. 2017).

Biosynthesis of  $\alpha$  and  $\beta$ -pinene via geranyl pyrophosphate through a soluble enzyme system from sage was carried out by (Gambliel and Croteau, 1982); in fact, soluble enzyme preparations from the tissue of immature sage catalyzed the conversion of acyclic C<sub>10</sub> precursor geranyl pyrophosphate to  $\alpha$  and  $\beta$ -pinene majorly and to other monoterpene olefins to lesser amounts.

Gastrointestinal transit is when the food leaves the stomach and passes through the intestines. To treat malfunctions related to this transit, mono-terpene-containing medicinal plants are used, especially plants that have  $\alpha$  and  $\beta$ -pinene (Falcão et al. 2008; Schmeda-Hirschmann and Yesilada 2005).  $\alpha$ -Pinene is an antibiotic resistance modulator for *Campylobacter jejuni* (Kovač et al. 2015). Also, results of the  $\alpha$ -pinene impact on pupal pain in Wistar mice showed that it could reduce pain at concentrations of (0.2 and 0.4  $\mu$ M) (George et al. 2015). Overproduction of reactive oxygen species (ROS) leads to impaired brain function. Studies have shown that  $\alpha$ -pinene prevents the production of intracellular ROS. Apoptosis is also reduced by  $\alpha$ -pinene. Therefore, it can be concluded that  $\alpha$ -pinene can defend the nervous system (George et al. 2015). Endocarditis is a disease caused by an infection of the heart wall or endocardium caused by various germs. Both  $\alpha$ - and  $\beta$ -pinene have shown inhibitory activity against bacterial strains tested and slowed the growth of endocarditis (Porres-Martínez et al. 2016). It also reversibly inhibits acetylcholinesterase, and thus has the potential to treat neurodegenerative diseases such as Parkinson's and Alzheimer's diseases (Risner et al. 2020).

Plants create large amounts of VOCs that protect them against biotic agents like viruses and their vectors. These metabolites may be linked to the susceptibility/tolerance of citrus species to the tristeza virus (CTV), a major biotic limitation in the citrus sector. The VOCs pattern from the leaves of three CTV-tolerant varieties, such as Carrizo citrange, *Citrus volkameriana*, and Forner-Alcaide no. 5, was compared to a CTV-susceptible citrus variety, including *Citrus aurantium* (Guarino et al. 2021). The released VOCs were evaluated using headspace SPME, whereas the leaf metabolites were examined using heptane extraction and GC-MS. Most of the VOCs released and sequestered in the leaves of CTV-tolerant, and susceptible citrus types were

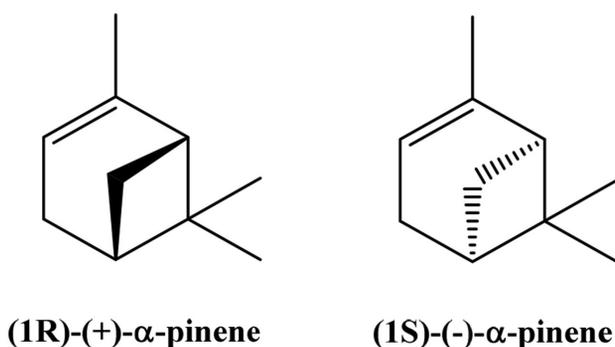


Figure 1. Chemical structures of  $\alpha$ -pinene.

volatile terpenes (VTs). The analysis of VOCs in the leaf tissues of CTV-tolerant varieties revealed a higher presence of monoterpenes, including  $\alpha$ -pinene, p-cymene, and limonene. These components have been identified with antiviral and insect repellent properties. A quality control study was conducted on *F. gummosa* oleo-gum resin extracted from standard plants and commercial samples from various parts of Iran (Moein et al. 2021). EO, standard fruit EO, and dichloromethane extract were obtained and evaluated using GC/MS. The samples were compared qualitatively through high-performance thin-layer chromatography (HPTLC). GC/MS analysis identified  $\beta$ -pinene,  $\alpha$ -pinene, and  $\delta$ -3-carene in commercial, EO and  $\beta$ -pinene,  $\delta$ -3-carene, and  $\alpha$ -pinene in standard samples, as significant components, respectively.

### Conventional extraction of $\alpha$ -pinene

Two types of techniques including conventional and novel methods could be used for the extraction of  $\alpha$ -pinene (Table 1); each extraction method is described in the following sections. Illustrations of these techniques are presented in Figure 2.

### Distillation

#### Hydrodistillation (HD)

HD is a standard and simple method used to extract compounds and EOs from plant components such as flowers, stems, or wood. In this method, natural water-insoluble products with high boiling points are separated. One of the advantages of this method is that the extracted oils are partially protected because the water around the extracted compounds acts as a barrier to prevent overheating. Another advantage of this method is that the target compounds are distilled at  $<100^{\circ}\text{C}$  (Matin et al. 2021). HD involves packing plant materials in a still compartment, adding enough water, and then bringing the mixture to a boil. It is also possible to inject direct steam into the plant samples. HD was used to extract

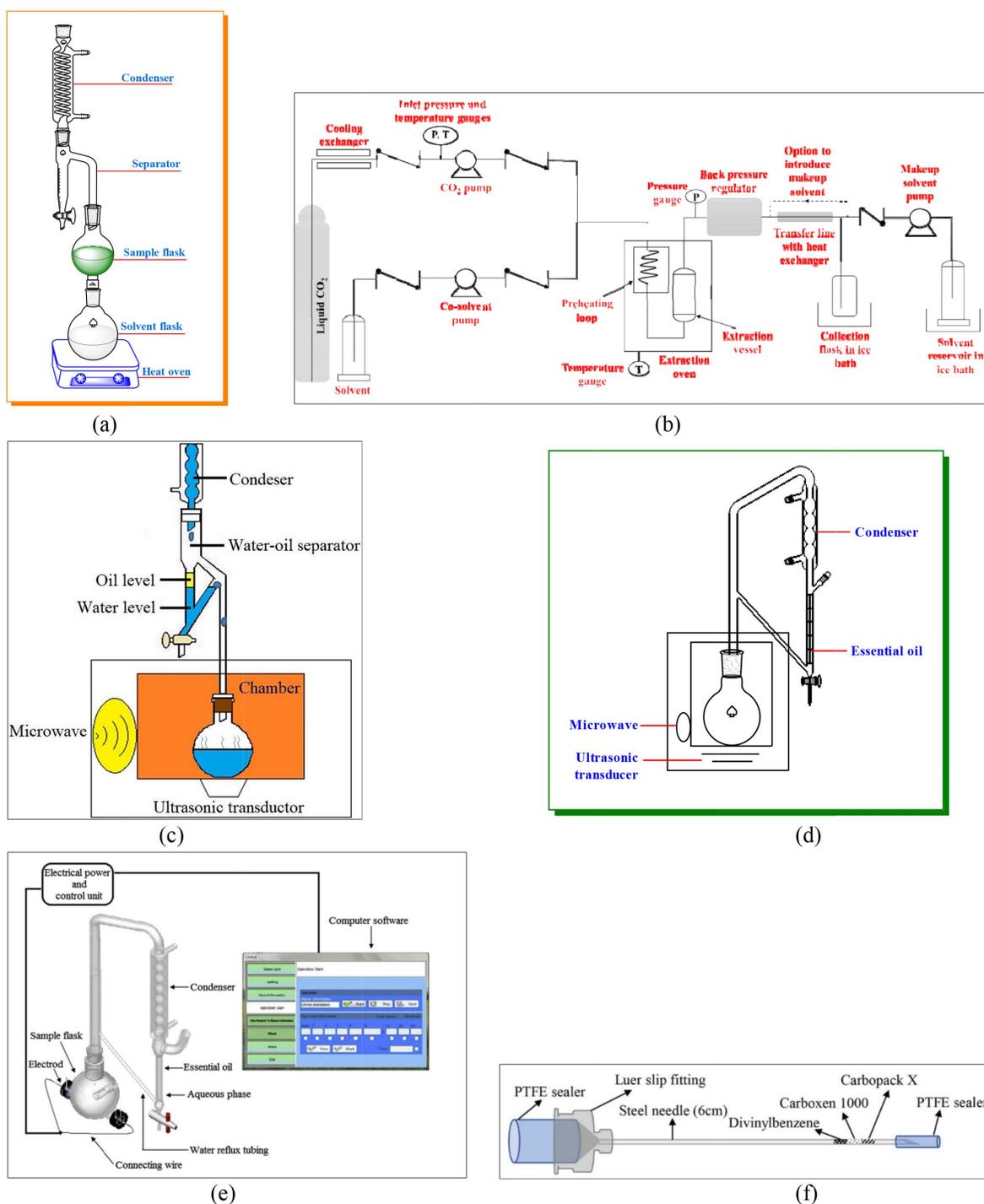
EOs from the *Myrtus communis* L. aerial portion, yielding 0.87% EOs (Zine Laabidine et al. 2021);  $\alpha$ -pinene (12.22%) was one of the major components. A total of nine samples from various locations around Sicily were analyzed (Siracusa et al. 2019). The metabolite content of leaves and myrtle berries were analyzed using cascade extraction and a single hydroalcoholic extraction approach, respectively, while EOs were extracted from leaves and berries using HD processes (100g of air dried leaves and 200g berries were subjected to HD process according to European Pharmacopoei for 3h). The presence of various polyphenols, particularly highly hydroxylated flavonols like quercetin, myricetin, and ellagic acid, was found in all of the extracts. Analysis of EOs was performed using Gas chromatography-mass spectrometry (GC-MS) and GC with flame-ionization detection (GC-FID). In total, 34 components were identified with  $\alpha$ -pinene, linalool, linalyl acetate, myrtenyl acetate, and 1,8-cineole, dominating.

The amount of  $\alpha$ -pinene extraction by HD method in some plants depends on the different habitats, parts plant, and daylight time. Different ecotypes and plant types also affect the amount of EO extracted and the amount of  $\alpha$ -pinene extracted. Among 23 ecotypes of Ajowan, although it was planted in the field in one month, the percentage of  $\alpha$ -pinene extraction for ecotypes was different, with a minimum of 0.05% and a maximum of 0.34%, and also percentage extraction of EO differed from 2.7% to 6.1% (Mirzahosseini et al. 2017). Even the type of culture in this method affects the yield of EO and the extraction of target compounds. For example, in comparing two types of hydroponic and pot cultures on *Petroselinum crispum* (Mill.), the EO extraction efficiency was better in hydroponic cultivation, and the amount of  $\alpha$ -pinene was 38.4 mg/L (Rattan et al. 2022).

Another parameter of  $\alpha$ -pinene extraction is the air velocity dryer changes before the extraction process. Other factors affecting the extraction rate of  $\alpha$ -pinene in the HD method are temperature and storage time before extraction. In 2018, Mohtashami et al. (2018) put the EO compounds of the *Satureja hortensis* L plant in three levels of storage temperature

**Table 1.** Difference methods for the extraction of  $\alpha$ -pinene.

| Conventional extraction methods                           | Novel extraction methods  |
|---|---|
| Distillation  | Supercritical fluid extraction (SFE)  |
| Hydrodistillation (HD)                                    | Ultrasound-assisted extraction (UAE)  |
| Enzyme-assisted extraction combined with HD (EAE with HD) | Headspace solid-phase microextraction (HS-SPME) or Ultrasound-assisted headspace solid-phase microextraction (UA-HS-SPME) |
| Steam distillation (SD)                                   | Microwave-assisted extraction (MAE)   |
| Solvent extraction  | Microwave-assisted hydrodistillation (MAHD)   |
| Cold press method   | Solvent-free microwave extraction (SFME)  |
| Vacuum-assisted sorbent extraction (VASE)                 | Ultrasonic-microwave-assisted extraction (UMAE)   |
|   | Ultrasonic/microwave-assisted hydrodistillation extraction (UMHE)   |
|   | Ohmic-assisted hydrodistillation (OAHD)   |
|   | Gas-liquid microextraction (GLME)   |
|   | Needle trap microextraction (NTME)  |
|   | Indirectly suspended droplet microextraction (ISDME)  |
|   | Purge and Trap (P&T)  |
|   | Simultaneous distillation-extraction (SDE)  |
|   | Acidic and enzymatic pretreatment via distillation and UAE  |
|   | Steam explosion method  |
|   | Cold centrifuges method   |
|   | Electromagnetic induction heating (EMIH)-assisted extraction  |
|   | Hybrid solar distillation system  |
|   | Combination of solid phase microextraction (SPME) and solvent-assisted flavor evaporation (SAFE)                          |
|   | Headspace stir-bar sorptive extraction  |



**Figure 2.** Different extraction methods of  $\alpha$ -pinene including (a) Steam distillation (SD), (b) Carbon dioxide expanded ethanol (CXE) and Supercritical fluid extraction (SFE), (c) Ultrasonic-microwave-assisted extraction (UMAE), (d) Ultrasonic/microwave-assisted hydrodistillation extraction (UMHE), (e) Ohmic-assisted hydrodistillation (OAHD) apparatus, and (f) Needle trap device.

before extraction; room temperature (21 °C), refrigerator temperature (4 °C), and freezer temperature (−20 °C). Storage time (no, three, and six months' storage) were tested and reported to decrease at each of the three storage temperatures of  $\alpha$ -pinene and carvacrol precursors. However, the carvacrol content increased significantly after 6 months, and also, the changes in the  $\alpha$ -pinene amount in different storage conditions were significant only at −20 °C so that the lowest amount of  $\alpha$ -pinene (0.72%) at freezer temperature (after 6 months' maintenance) was observed (Mohtashami et al. 2018).

Karami et al. (2017) studied the effect of drying temperature and air velocity on *Mentha aquatica* L. EO content; they, before extraction with Clevenger-type apparatus, dried the aerial parts of the plant at temperatures of 40–60 °C and airflow of 1 and 2 m/s, and concluded that in HD method,  $\alpha$ -pinene extraction also depends on the air-drying speed, and increasing air velocity dryer of 1–2 m/s caused increasing  $\alpha$ -pinene percentage. Schimitberger et al. (2018) reported that the yield of EO from *Schinus terebinthifolia* ripe fruit (for the HD extraction) was twice that of the unripe fruit ones. In the ripe fruit,  $\alpha$ -pinene

and  $\delta$ -3 carene are possible chemical markers of this species, while limonene can be used as a substance characteristic of unripe fruit. In this study, the extraction rate of  $\alpha$ -pinene was 14.9% in the ripe fruit.

Even the color and density of the EO affect the  $\alpha$ -pinene content of some plants. According to the results of Gonçalves et al., the extract of *Piper callosum*, which contained a light yellow EO with less density than water, included 19.2%  $\alpha$ -pinene while colorless EOs with a density higher than water had no amount of  $\alpha$ -pinene (Gonçalves et al. 2019). Healthy and aphid-infected leaves are also effective in  $\alpha$ -pinene levels of some plants. In 2019, Harouak et al. reported that *Atlantic pistachios*, *Pistacia atlantica* Desf, were infected by *Geoica swirskii* and *Forda riccobonii*, foliar galls-inducing aphids. They noted that the infected leaves were rich in  $\alpha$ -pinene (Harouak et al. 2019).

Capturing EO at sequential timeframes during HD with water produces fractions containing unique compounds and antioxidant capacity. According to the results obtained from sequential timeframes during HD in 5 min intervals in the range of 0–240 min for three plants (*Juniperus Virginiana*, *Juniperus Excelsa*, and *Juniperus Sabina* Osabina)  $\alpha$ -pinene for *Juniperus Excelsa* plant was reported to be the highest amount (34–36%) at a fraction of 0–5 min. In the other plants, sequential timeframes of 0–5 min were > different sequential timeframes for  $\alpha$ -pinene (Semerdjieva et al. 2019). The efficiency of EO extraction and extraction rate of  $\alpha$ -pinene in HD is very variable. For example, the literature states that the water distillation method for extracting  $\alpha$ -pinene from *Xylopiya aethiopicum* fruit required 4 h at 95°C, and after drying, the EO was kept at 4°C. The EO extraction efficiency was 4.2%, and the extraction rate of  $\alpha$ -pinene was reported at 7.4% (Tegang et al. 2018). One of the highest levels of  $\alpha$ -pinene extracted by the HD method is related to *Teucrium polium*. The EO of this plant was extracted for 3 h, and the EO extraction efficiency was 75%, and the  $\alpha$ -pinene extraction rate was 99.7%, which was the highest extraction rate (Mahalleh, Zamein, and Dahmardeh 2020).

#### Enzyme-assisted extraction (EAE) combined with HD

EAE is a process that disrupts the cell wall via biodegradation under mild conditions. The enzymes of endoglycanases, xylanases, and amyloglycosidases have also been used to destroy the cell wall of plants, which, in turn, lead to the extraction of EOs and  $\alpha$ -pinene (dos Santos Reis et al. 2020). One of the advantages of EAE is the release of more oil from the plant, and the lack of additional equipment is another benefit of EAE (Kurmudle et al. 2013; Li et al. 2012; Vladić et al. 2022). In one study, Rashmi et al. (2017) investigated the effect of EAE on the composition of *Cymbopogon citratus* leaf volatile oil. EAE with  $\beta$ -galactosidase and cellulase produced 0.82 and 0.91% of volatile oil, respectively. In contrast, with a mixture of enzymes (cellulase and  $\beta$ -galactosidase), the yield of volatile oil was 0.97% and extraction efficiency in conventional HD was 0.4%. It was noteworthy that in the EAE with each of the enzymes, some important compounds, such as  $\alpha$ -pinene, were not extracted separately, while in the EAE using a mixture of  $\beta$ -galactosidase and cellulase, the amount of  $\alpha$ -pinene was obtained to be 0.4% (Rashmi et al. 2017).

#### Steam distillation (SD)

In this method, the temperature of 100°C is used, and the time varies from 1 to 4 h. The sample is dried over anhydrous  $\text{Na}_2\text{SO}_4$  and stored under refrigeration at +4°C or –18°C. In addition, in this method, different devices have been used, and the amount of EO and  $\alpha$ -pinene extraction also varies according to the conditions. For example, to extract EO and  $\alpha$ -pinene from *Tetraclinis articulata* and *Juniperus phoenicea* plants, Clevenger type has been used and EO yields were 0.57 and 1.67% of dry weight and extraction amounts of  $\alpha$ -pinene were reported to be 9.3 and 48.2 percent, respectively (Harmouzi et al. 2016).

In another case, the EO was obtained in a pilot-scale equipment, which has a boiler, an extraction vessel, a liquid–liquid separator, and a heat exchanger for the *Achyrocline satureioides* plant, and in this method, four different pressures were used. EO total yield obtained at 1.0, 1.5, 2.0 and 2.5 bar were 0.082, 0.061, 0.069, and 0.074 g oil/100 g plant, respectively. The mathematical model used in this study represented yield versus time in the experimental curves at the four different pressures very well. The researchers of that study also concluded that the relation between the concentration of the compound and the aromatic intensities is not directly proportional, except for  $\alpha$ -pinene, which presented a high concentration and aromatic intensity (Pires et al. 2019). In the case of SD and the use of boiler, in addition to the extraction time and pressure, the particle size is also involved in the extraction of  $\alpha$ -pinene. For the extraction of  $\alpha$ -pinene from *Myrtus communis* L., the optimum conditions were selected: distillation duration 75 min, boiler occupancy rate 100%, and particle size 20 mm. Under these conditions, the rate of  $\alpha$ -pinene extraction was reported to be 33.1% (Kaya et al. 2020). Another optimization for SD was reported using flask, separator, oven, and condenser equipment, as shown in Fig. 2a (Quyen et al. 2020). The mixing ratio of lemon juice and peel was reported to be 3:1 (mL/g). In these conditions, the highest EO yield was obtained at 2.1% and  $\alpha$ -pinene at 2.1%.

Studies on the pilot plant scale showed that SD method results in the enhancement of some EO compounds (in the plant) and the diminishment of some other ones; for example, SD was investigated for pre-domesticated Spanish *S. montana*, and the results showed that SD increased the EO content in  $\alpha$ -pinene,  $\alpha$ -thujene, p-cymene,  $\alpha$ -terpinene, and transcaryophyllene, while decreased thymoquinone thymol, borneol, and  $\beta$ -bisabolene (Navarro-Rocha et al. 2020). The fruiting period of the plant also affects the extraction of EO and  $\alpha$ -pinene. Garzoli et al. reported in a study that *Foeniculum vulgare* Miller had a significant increase in the amount of EO up to five times during the fruiting period of October. In this method, SD was used, and the oil was collected at different time intervals (1, 2, 3, 6, 12, and 24 h) (Garzoli et al. 2018). In the SD method, it was reported that the date of cultivation and organic fertilization affects the production and composition of EOs obtained from *Ocimum basilicum* and the amount of  $\alpha$ -pinene (Omer et al. 2016). In Table 2, several studies focused on the distillation techniques for extracting  $\alpha$ -pinene are listed. According to Table 2, the time of HD is about 1–8 h.

**Table 2.** Research conducted on the extraction of  $\alpha$ -pinene using various distillation methods.

| Fruit/plant   | Extraction time (h) | Yield of $\alpha$ -pinene (%) | Origin                          | Region/collect/grown or harvest time                            | References                             |
|---|---------------------|-------------------------------|---------------------------------|---|--|
| 23 ecotypes belonging to Ajowan.                    | 4                   | 0.05–0.34                     | Seeds                           | Tehran, Iran/October 2014                                       | Mirzahosseini et al. (2017)            |
| <i>Xylopia aethiopica</i>                           | 4                   | 7.4                           | Dried fruits                    | Bafoussam (Cameroon)/July 2015                                  | Tegang et al. (2018)                   |
| <i>Teucrium polium</i>                              | 3                   | 12.5                          | Leaves                          | Sistan and Baluchistan, Iran/April 2018                         | Mahalleh, Zamein, and Dahmardeh (2020) |
| <i>Ferulago angulata</i>                            | 3                   | 14.6–34.4                     | Aerial parts                    | Kurdistan, Kermanshah and Lorestan, Iran/May–July 2014          | Shahbazi (2016)                        |
| <i>Myrtus communis</i> L.                           | –                   | 32–65.1                       | Leaves                          | Baghdad, Iraq   | Sabri et al. (2016)                    |
| <i>Astrodaucus persicus</i>                         | 4                   | 20.9                          | Aerial parts                    | Kurdistan Iran/September 2010                                   | Goodarzi et al. (2016)                 |
| <i>Rosa brunonii</i> Lindl                          | 4                   | 5.7                           | Fresh flowers                   | Uttarakhand-India/April 2009                                    | Verma, Padalia, and Chauhan (2016)     |
| <i>Croton jacobinensis</i> Baill                    | 2                   | 4.8                           | Stalks                          | Ceará, Northeast of Brazil/April 2013                           | Pinto et al. (2016)                    |
|   | 2                   | 7.9                           | Leaves                          |   |  |
|   | 2                   | 10.9                          | Inflorescences                  |   |  |
| <i>Artemisia arborescens</i> L.                     | 3                   | 0.02                          | Aerial parts                    | Messina, Italy/January 2014                                     | Costa et al. (2016)                    |
| <i>Pinus taeda</i> L.                               | 1-2                 | 0.25                          | Fresh needles/twigs             | Paraná, Brazil/April 2011                                       | Teixeira et al. (2016)                 |
|   | 1-2                 | 11.3                          |                                 |   |  |
| <i>Lavandula stricta</i>                            | –                   | 58.3                          | Aerial parts                    | Genow, Hormozgan, Iran  | Alizadeh and Aghaee (2016)             |
|   | –                   | 63.5                          |                                 | Rodan, Hormozgan, Iran  |  |
| <i>Croton sonderianus</i>                           | 2-4                 | 3.5                           | Leaves                          | Pernambuco-Brazil/July to August 2012                           | Souza et al. (2017)                    |
|   | 2-4                 | 6                             |                                 | Pernambuco-Brazil/Feb. to April 2013                            |  |
| Rosemary  | 3                   | 18.6                          |                                 | Tehran/Spring   | Raeisi et al. (2017)                   |
|   | 3                   | 32.4                          |                                 | Kerman/Spring   |  |
| <i>Croton grewiioides</i> Baill                     | 4                   | 47.4                          |                                 | Serra Branca-Brazil   | de Medeiros et al. (2017)              |
| <i>Boswellia dalzielii</i>                          | 4                   | 15.2                          |                                 | Ségbana region (Benin)/Dec. 2012                                | Kohoude et al. (2017)                  |
| Three <i>Ocotea</i> species                         | 3                   | 9.8–22.5                      | Leaves/branches                 | Caxiuanã National Forest, Brazil                                | Da Silva et al. (2017)                 |
| <i>Eupatorium intermedium</i>                       | –                   | 22.6                          | Flower                          | Palmeira (Paraná, Brazil)                                       | Czaikoski et al. (2017)                |
| <i>Rudbeckia triloba</i>                            | 4                   | 46                            | Dried leaves                    | Bucharest, Romania/August 2016                                  | Moldovan et al. (2017)                 |
|   | 4                   | 40.1                          | Flower                          |   |  |
| <i>Cymbopogon khasianus</i>                         | –                   | 0.891                         | Leaves                          | Meghalaya, North East India/June 2013                           | Lal et al. (2018)                      |
| <i>Tetraclinis articulata</i> (Vahl)                | 3                   | 38.7                          | Branches                        | Forest of a region in East Morocco April (2016)                 | Sadiki et al. (2018)                   |
| <i>Artemisia arborescens</i>                        | 4                   | 1.9                           | Leaves and                      | 55 Sites during   | Fanouriou et al. (2018)                |
| <i>Cistus salviifolius</i>                          | 4                   | 0.35                          | inflorescences                  | Flowering-Turkey/March, April, May, June, July, and August 2014 |  |
| <i>Crithmum maritimum</i>                           | 4                   | 0.87                          |                                 |   |  |
| <i>Ferula communis</i>                              | 4                   | 1.5                           |                                 |   |  |
| <i>Hypericum hircinum</i>                           | 4                   | 0.95                          |                                 |   |  |
| <i>Hypericum empetrifolium</i>                      | 4                   | 37.5                          |                                 |   |  |
| Ten populations of <i>Juniperus excelsa</i> complex | 5                   | 12.5–90.9                     | Leaves                          | Iran/November and December, 2006                                | Hojjati, Sereshti, and Hojjati (2019)  |
| <i>Cupressaceae</i> species                         | 3                   | 16.4–43.1                     | Dry leaves                      | Northwestern Algeria/autumn of 2016                             | Boufares, Hassani, and Alem (2019)     |
| White pepper ( <i>Piper nigrum</i> L.)              | –                   | 5.4                           | Seeds                           | Kien Giang, Vietnam   | Tran et al. (2019)                     |
| <i>Schinus terebinthifolius</i> Raddi               | 1-8                 | 7.03–9.2                      | Ripe fruits                     | Aracruz, Brazil/June 2014                                       | Guzzo da Silva et al. (2019)           |
| Rosemary  | 4                   | 35.5                          | Young leaves, branches, flowers | Lam Dong, Vietnam   | Ngân et al. (2019)                     |
| <i>Rosmarinus officinalis</i>                       | 3                   | 3.08–9.98                     | Leaves                          | Jerada and Taourirt, Morocco                                    | Sabbahi et al. (2019)                  |
| <i>Helichrysum italicum</i> (Roth) G. Don           | –                   | 28.50                         | Flower heads                    | Botanical Garden/June 2017                                      | Gismondì, Di Marco, and Canini (2020)  |
| Kaffir lime ( <i>Citrus hystrix</i> D.C)            | 1.5                 | 2.8                           | Fresh peels                     | Giang, Vietnam  | Le et al. (2020)                       |
| <i>Eucalyptus globulus</i>                          | 0.5-2.15            | 17.86                         | Leaves                          | Tra Vinh, Vietnam   | Ngo, Tran, and Le (2020)               |
| <i>Halophyte Echinophora spinosa</i> (Apiaceae)     | –                   | 13.8                          | Aerial parts                    | Corsica Island, France/September 2017                           | Pavela et al. (2020)                   |
|   | –                   | 0.2                           | Root                            |   |  |
| Vietnamese green pepper ( <i>Piper nigrum</i> )     | 3                   | 4.45                          | Seeds                           | Vung Tau, Vietnam   | Dao et al. (2020)                      |
| <i>Ferulago cassia</i> Boiss                        | 3                   | 7.6-12.4                      | Fruits                          | Lakes Region, Turkey/2018                                       | Sanli et al. (2020)                    |
| <i>Myrtus communis</i>                              | 3                   | 19.39                         | Leaves                          | Ouazzane, Morocco/June 2015                                     | El Hartiti et al. (2020)               |
| <i>Marrubium heterodon</i>                          | 3                   | 17.3                          | Aerial parts                    | Niğde, Turkey   | Arabacı et al. (2020)                  |
| <i>Tetraclinis articulata</i>                       | 3                   | 8.1-21.5                      | Flower                          | Tunisia, North Africa/2013                                      | Rguez et al. (2020)                    |
| <i>Eugenia punicifolia</i>                          | 3                   | 49.7                          | Leaf                            | Maranhão, Brazil/May 2014                                       | Fernandes et al. (2021)                |

(Continued)

Table 2. (Continued)

| Fruit/plant  | Extraction time (h) | Yield of $\alpha$ -pinene (%) | Origin               | Region/collect/grown or harvest time                        | References                                    |
|--|---------------------|-------------------------------|----------------------|---|---|
| <i>Eugenia punicifolia</i>   | 3 h                 | 49.7                          | Aerial parts         | Parque Nacional Nascentes do Rio Parnaíba, Maranhão, Brazil | Fernandes et al. (2021)                       |
| <i>Myrciaria tenella</i>   | 5                   | 19.4                          | leaves               | Rio de Janeiro, Brazil/August 2018                          | Gonçalves et al. (2021)                       |
| <i>Rhipicephalus sanguineus</i> ,<br>Ten populations of<br><i>Baccharis dracunculifolia</i> DC | over 2              | 13.8                          |                      | Brazil/July 2018  | Rezende et al. (2021)                         |
| Rosemary varieties<br>(Boule, Gori, Joyce,<br>Vicom, Jord)                                     | 2.5                 | 2.02-4.6                      | Branches with leaves | Paraná, south of Brazil/March 2016                          | Tomazzoli et al. (2021)                       |
| Fennel   | 2                   | 3.1-37.3                      | Dried flowering tops | Sanremo, Italy  | Oualdi et al. (2021)                          |
|  | 3.5 and 6           | 22.46                         | Umbels               | Blida, Algeria/harvested in November 2015                   | Dahmani et al. (2022)                         |
|  | 3.5 and 6           | 1.3                           | Seeds                |   |   |
| <i>Satureja montana</i> L.   | 2                   | 3.1                           | Leaves               | Lavras, MG, Brazil  | Rezende et al. (2022)                         |
| <i>Myristica fragrans</i> H.   | 2                   | 13.8                          | Seed                 |   |   |
| Sweet Orange <i>Citrus sinensis</i> Osbeck   | –                   | 0.2                           | Fruits               | Different geographical regions of Nepal                     | Bhandari et al. (2021)                        |
| Mandarin <i>Citrus reticulata</i> Blanco   |                     | 0.57                          |                      |   |   |
| Pummelo <i>Citrus grandis</i> Osbeck Red Flesh   |                     | 0.58                          |                      |   |   |
| Pummelo <i>Citrus grandis</i> Osbeck White Flesh   |                     | 0.42                          |                      |   |   |
| Lamiaceae<br>( <i>Dracocephalum kotschyi</i> Boiss.)   | 3 h                 | 13.7                          | Aerial parts         | Daran (Isfahan, Iran)                                       | Ghavam et al. (2021)                          |
| Coriander  | –                   | 6.1                           | Seeds                | South Indian spices   | Ashokkumar et al. (2021)                      |
| Cumin  |                     | 0.76                          |                      |   |   |
| Fennel   |                     | 0.46                          |                      |   |   |
| Indian mustard   |                     | –                             |                      |   |   |
| White Pepper ( <i>Piper nigrum</i> L.)   | 96 min              | 5.2                           | Seeds                | Phu Quoc Island, Kien Giang, Vietnam                        | Tran et al. (2020)                            |
| <i>Pistacia atlantica</i> oleoresin (PAO)  | 5 h                 | 18.1                          | Oleoresin            | Oramanat (Kurdistan, west of Iran)                          | Bagheri et al. (2019)                         |
| <i>Lepechinia heteromorpha</i>   | 4 h                 | 1.2                           | Leaves               | Ecuadorian Andean   | Gilardoni et al. (2019)                       |
| Apiaceae ( <i>Prangos ferulacea</i> (L.) Lindl.)   | 4 h                 | 22.1                          | Aerial parts         | Khoy, Northwest of Iran)                                    | Delnavazi et al. (2017)                       |
| <i>Dracocephalum kotschyi</i> Boiss.   | 3 h                 | 12.6                          |                      | Sari, North of Iran   | Golparvar et al. (2016)                       |
| <i>Ferulago angulata</i>   | 2 h                 | 10                            | Fruits               | Kurdistan, west of Iran                                     | Shafaroodi, Roozbahani, and Asgarpanah (2021) |
| <i>Tetraclinis articulata</i>  | 3                   | 9.3                           | Leafy twigs          | M'irt, Morocco/June 2012                                    | Harmouzi et al. (2016)                        |
| <i>Juniperus phoenicea</i>   | 3                   | 48.2                          |                      | Tounfit, Morocco/April 2012                                 |   |
| Lavender EO  | 3                   | 0.1–0.5                       | Aerial parts         | Northern Italy  | Grandi et al. (2016)                          |
| Rosemary EO  | 3                   | 14.4–34.3                     |                      |   |   |
| Thyme EO   | 3                   | 0.07–1.4                      |                      |   |   |
| Abies Koreana twigs  | 1.5                 | 12.9                          | Twigs                | Inje, Gangwon-do, Korea/April 2015                          | Seo, Sowndhararajan, and Kim (2016)           |
|  | 1.5                 | 12.8                          |                      | Inje, Gangwon-do, Korea/June 2015                           |   |
|  | 1.5                 | 11.05                         |                      | Inje, Gangwon-do, Korea/August 2015                         |   |
|  | 1.5                 | 13.02                         |                      | Inje, Gangwon-do, Korea/Oct 2015                            |   |
| <i>Juniperus foetidissima</i>  | 1                   | 2.66                          | Fruit                | Arasbaran-Iran/August                                       | Parvin Salehi, Mirza, & Calagari, (2016)      |
| <i>Juniperus oblonga</i>   | 1                   | 22.04                         |                      |   |   |
| <i>Hypericum perforatum</i> L.   | 4                   | 30.9                          | Inflorescences       | Lipova, Arad-Romania/July 2015                              | Moleriu et al. (2017)                         |
| Kaffir lime ( <i>Citrus hystrix</i> DC)  | 3.5                 | 35.5                          | Fresh peels          | Giang, Vietnam  | Ngan et al. (2019)                            |
| <i>Lantana camara</i>  | –                   | 3.3                           | Leaves               | Faisalabad, Pakistan  | Sajid et al. (2021)                           |
| <i>Rosmarinus officinalis</i> L.   | 2–6                 | 22.1                          |                      | Highland, Vietnam   | Nguyen et al. (2022)                          |
| Juniper species  | 3                   | 14.9–34.9                     | Leaves/small twigs   | Rhodope Mountains, Bulgaria/June                            | Semerdjieva et al. (2021)                     |

### Solvent extraction

Oxygenated molecules have more significant distribution coefficients than  $\alpha$ -pinene, resulting in solvent selectivity  $> 1.0$ . Compared to eucalyptol, camphor exhibited higher solvent selectivity and distribution coefficients. Low phase solubility was caused by high water content in the solvent, resulting in higher solvent selectivity but lower distribution coefficients. Improved oxygenated molecules in the systems, on the other hand, resulted in increased higher distribution coefficients and solubility but poorer solvent selectivity.

Liquid–liquid extraction (LLE) is a standard ternary system, also known as solvent extraction, that separates chemicals based on the different solubility of the solute chemical in the two solvents (Huang, Ramarao, and Ramaswamy 2013). LLE processes have been widely used to extract active pharmaceutical compounds at both the laboratory and industrial scales (Li et al. 2022). In a study, four solvents including dichloromethane, chloroform, *n*-hexane, and ethyl acetate were used to determine the target compounds in rose aromatic water extract. According to the obtained results, for the extraction of target compounds including  $\alpha$ -pinene,  $\alpha$ -humulene, linalool, methyl eugenol,  $\beta$ -caryophyllene, and eugenol, and so on, in general, *n*-hexane was the worst LLE solvent and the amounts of  $\alpha$ -pinene obtained for chloroform, dichloromethane, ethyl acetate, and *n*-hexane solvents were reported to be 25.8%, 32.1%, 57.5%, and 32.7%, respectively (Canbay 2017).

Solid–liquid extraction (SLE) is one of the most widely used methods for solid samples. It can be described as an extraction process consisting of desorption from the surface of the solid sample, diffusion within the sample matrix, and partitioning/solvation to the extraction phase. Slow extraction speed and time consuming process completion are its disadvantages. In one study, oleic gum resin was extracted by the SLE method in two modes: batch-mode and continuous-flow. In the batch-mode, 3 g powder oleo-gum resins Hoojri was extracted with 15 mL EtOH in the dark, and extraction was carried out at 550 rpm for 2.5 h. The extract was filtered, and the residue was subjected to the extraction with the same volume of ethanol. In the continuous-flow SLE, EtOH was used as a solvent, and a high-pressure liquid chromatography (HPLC) pump connected to a preheating coil as well as an extraction vessel inside a heated GC furnace were used. According to the results, the amount of  $\alpha$ -pinene obtained in the continuous method was  $>$  the batch, and the amount of  $\alpha$ -pinene in the continuous method was 21.1, and in the batch method was 14.9% (Al-Hamimi et al. 2016).

The accelerated solvent extraction process uses high pressure and temperature, resulting in the extraction taking less time, needing less solvent, and offering improved analyte recovery. An accelerated solvent extractor was used to extract bioextracts from plant shoots of *Cistus ladanifer* L. using *n*-hexane (Santos et al. 2017). Some valuable organic compounds such as  $\alpha$ -pinene, camphor, camphene, verbenone, and fenchone were identified and quantified using GC-MS. São Domingos bioextracts contained the highest concentration of  $\alpha$ -pinene while the other valuable

constituents were similarly concentrated. There was no correlation between potentially hazardous elements in the shoots and bioextract composition.

A study was conducted to develop a green oleo-extract of all nonvolatile and volatile (e.g.  $\alpha$ -pinene) bioactive substances from rosemary leaves to a waste-free biological concept of bio-based solvents made from vegetable oils and their amphiphilic derivatives (Li et al. 2019). Adding oil derivatives to soybean oils boosted the oleo-extraction of nonvolatile antioxidants by 66.7%, and were effective on solvating volatile aromatic compounds in refined soybean oils by 16%. Analyses of resin mass flow rate were used to identify adult slash pine (*Pinus elliottii* Engelm.) trees with high resin yield in a short timescale to develop elite forests for tapping before it began (Neis et al. 2019). The ratio of  $\alpha$ -/ $\beta$ -pinene in resin monoterpenes was lower in trees that gained more yield, and these trees also possessed more limonene in total terpenes than trees with lower yields.

An imidazolium-based ionic liquid  $[C_2C_1\text{Imim}][\text{OAc}]$  has been used to extract volatile terpenes (i.e.  $\alpha$ -pinene) from loblolly pine (Papa et al. 2017). Three types of pine tissue were investigated for potential sugar extraction and  $\alpha$ -pinene recovery after treatment with IL. Using an ionic liquid for the simultaneous extraction of  $\alpha$ -pinene and processing of wood to produce a carbohydrate-rich component susceptible to fermentable sugars by enzymatic hydrolysis is a promising idea. The  $\alpha$ -pinene extracted using the IL method was remarkably similar to the  $\alpha$ -pinene extracted using the traditional hexane method.

### Cold press along with the vacuum-assisted sorbent extraction (VASE)

Another name for the cold pressing method is scratching. The EOs of fruits such as lemons, citrus fruits, flowers, and medicinal plants can be extracted in this way. In this process, the desired compounds are extracted by rubbing the outer layer of plants (Geramitcioski, Mitrevski, and Mijakovski 2018). Due to the adverse effects of high temperature on extraction processes, one of the critical merits of this method is low-temperature extraction in which the compounds are first compressed and, then, filtered at low temperatures.

VASE is a unique method that uses a vacuum to extract sorbents from headspace. Utilizing vacuum to boost the yield of complex chemicals to extract, in conjunction with marketed sorbent traps, has been demonstrated (Jeleń, Gaca, and Marcinkowska 2018). In addition, a significant amount of Tenax is utilized to fill the sorbent traps (About 500 times the typical SPME volume), which aids in the extraction process by allowing exhaustive extraction. Furthermore, because VASE uses closed traps and vacuum to drive extraction, there are no concerns with breakthrough volumes commonly seen for P&T systems and other dynamic headspace approaches. To extract terpenes from coriander oil, the most important of which was  $\alpha$ -pinene, the cold press method was used along with the VASE method. The characteristics of the compounds extracted using VASE were

compared by classical water distillation. In the water distillation method, the amount of extracted  $\alpha$ -pinene was 12.79%, while in the VASE method, at 20 and 60 °C, the  $\alpha$ -pinene content was 17.9% and 11.99%, respectively, which indicates the positive effect of low temperature in these extraction methods (Jeleń, Marcinkowska, and Marek 2021).

## Novel extraction methods

### Supercritical fluid extraction (SFE)

The application of a solvent with properties intermediate between a liquid and gas is employed for more effective extraction of a solute from a solid matrix, which belongs to a technology of SFE. The advantages of this extraction are rapid release of solutes, increased extraction rate, and high solvent strength. This method is usually used to replace conventional industrial methods. The SFE method has significant properties for EO extraction and subsequently  $\alpha$ -pinene, including being non-flammable, non-corrosive, nontoxic, and non-explosive because of the lack of consuming risky solvents in this method. Because SFE system operates at low temperatures, therefore it prevents heat damage. The EO extracted by this method rarely needs further refining due to the quality of the extracted compounds (Jin et al. 2010). It also solves the problem of thermal degradation, is environmentally safe, avoids the use of solvent and, therefore, the presence of solvent traces in the extracts is minimal, and presents a low operational cost (Topala and Tataru 2016). One of the crucial advantages of this method is extraction efficiency. A comparison of HD and SFE-CO<sub>2</sub> methods on *Rosmarinus officinalis* L. showed that the extraction efficiencies of both fresh and dried EOs obtained by HD were statistically significantly < those obtained by SFE-CO<sub>2</sub> (Kessler et al. 2022).

Generally, with increasing the temperature, diffusion rates in extractions are enhanced. In one study on *Boswellia sacra* tree resin, feasibility of using carbon dioxide expanded ethanol (CXE) as the extraction phase was explored, targeting two medium-polar analytes,  $\alpha$ -pinene, and cis-verbenol in *Boswellia sacra* tree resin. Also, an extraction method with CXE was optimized, giving optimal conditions of 40 °C, 9.3 MPa, and 0.31 molar fraction of CO<sub>2</sub> in EtOH (Al-Hamimi et al. 2016). Eventually, CXE and the developed method were compared with SFE and conventional SLE method, showing that CXE enables faster and more efficient extraction than both SLE and SFE methods. In fact, the use of CXE and other CO<sub>2</sub>-expanded liquids in the preparation of samples without increasing the temperature shows excellent potential for elevating the extraction rate (Figure 2b) (Al-Hamimi et al. 2016).

In extracting EOs from *Artemisia annua* L, it was concluded that the combination of supercritical carbon dioxide (scCO<sub>2</sub>) and molecular distillation was an exemplary method for separating EOs;  $\alpha$ -pinene was a vital component of EOs and also purification <120 °C could produce high-quality EOs with 56.1% yield (Li et al. 2017). In one study, the EO was extracted from pine branches by scCO<sub>2</sub> and HD

methods. The results showed that the major volatile profiles, including  $\alpha$ -pinene, in scCO<sub>2</sub> extracts and water distillation were quite similar, and when temperature and scCO<sub>2</sub> density increased, experimental design results exhibited higher extraction yields (up to 6%) (Gaspar et al. 2020). In an experiment on *Pistacia lentiscus* L,  $\alpha$ -pinene (32%) was the main constituent of the EO extracted in Tunisia. Among the various investigations to extract the EO, it was concluded that the lowest average particle diameter, the highest pressure, and the lowest flow velocity provided the most elevated conditions for extracting the tested income (Aydi et al. 2020).

### Ultrasound-assisted extraction (UAE)

Another method to achieve  $\alpha$ -pinene is to extract the EO using UAE method. Essential parameters in this method are extraction temperature, extraction time, and solvent-to-material ratio. Research into the extraction of EOs from *Myristica fragrans* points to the same thing. In this study, it was concluded that with an optimum temperature of 41.9 °C and extraction time of 29.6 min, and solvent-to-material ratio of 61.374 mL/g, optimal extraction conditions were obtained (Poorhashemi, Arianfar, and Mohammadi 2020). Ultrasonic pretreatment affects the extraction efficiency at room temperature. In a study on citrus waste by-products, pretreatment was performed by the UAE method, and results showed that the pretreatment produced a maximum (33.3% increase) of EO in 15.7 min and a solvent-to-sample ratio of 3.2/1 and also the amount of  $\alpha$ -pinene obtained was reported to be 0.22% (Sandhu et al. 2021). The reason for ultrasonic pretreatment, which causes more extraction of EO, is the rupture of the cell wall, which is accompanied by increased mass transfer and the formation of small cavities (Cameron, McMaster, and Britz 2009; Vinatoru 2001).

In addition to the mentioned items that are effective in the UAE method, one of the critical cases in this extraction method is the maximum output power. For example, in a study of nutmeg, extraction was performed using ethanol. All extractions were carried out at room temperature for 10 and 25 min using UAE at 20% of maximum output power (60 W) and 40% of maximum output power (120 W), respectively. The results showed that  $\alpha$ -pinene and 52 other compounds were extracted in nutmeg oleoresin using two methods of maceration and UAE. Experimental results also showed that in the UAE method, high-quality nutmeg oleoresin was produced with good performance in 10 min and 40% of maximum output power (Morsy 2016). Also, in another study, the effect of UAE and solvents was investigated simultaneously with the aim of extracting phytochemical compounds in the leaf extract of *Syzygium polyanthum*. Regarding the  $\alpha$ -pinene extraction, the highest value was for *n*-hexane extract with 4.9% and then ethyl acetate extract with 1.068%. For methanolic extract, no amount of  $\alpha$ -pinene was reported. Therefore, the type of solvent is also effective in extraction quality in this way (Abd Rahim et al. 2017). For a more comprehensive overview of the extraction of  $\alpha$ -pinene based on carbon nitride SFE and UAE techniques, some relevant studies are summarized in Table 3 (Allawzi,

**Table 3.** List of various studies designed to extract  $\alpha$ -pinene using SFE and UAE techniques.

| Technique          | Fruit/plant   | Solvent/temperature/<br>power  | Extraction time/<br>pressure   | Flow rate                              | Yield (%) | References                            |
|--------------------|---|--------------------------------|--------------------------------|--|-----------|---------------------------------------|
| SFE                | Algerian Rosemary   | 313 K                          | 3.5 h/22 MPa                   | 0.42 kg/h                              | 6.05      | Zermane et al. (2016)                 |
|                    | Angelicae dahuricae Radix   | 30, 40, and 50°C               | 1, 2 and 3 h/20, 25 and 30 MPa | –                                      | 6.25      | Sun et al. (2017)                     |
|                    | Roasted pistachio   | 70°C                           | 2 h/35 MPa                     | (50, 75 and 125 gCO <sub>2</sub> /min) | 24.47     | Barla Demirköz et al. (2018)          |
|                    | Jordanian Rosemary  | 35°C                           | 0.5 h/8.2 MPa                  | 100 mL/min                             | 55.10     | Allawzi, Allaboun, and Almasri (2019) |
|                    |   | 70°C                           | 0.5 h/8.6 MPa                  | 100 mL/min                             | 48.59     |                                       |
|                    |   | 55°C                           | 0.5 h/10.3 MPa                 | 100 mL/min                             | 44.41     |                                       |
|                    | Araçá ( <i>Psidium grandifolium</i> Mart. ex DC.)                     | 50°C                           | 6 h/20 MPa                     | 5 g/min                                | 20.75     | Bittencourt et al. (2019)             |
|                    | <i>Anethum graveolens</i> L. (dill) seeds                             | 40°C                           | 2 h/20 MPa                     | 25 L/h                                 | 6.43      | Li et al. (2021)                      |
|                    | Omija ( <i>Schisandra chinensis</i> (Turcz.) Baill.)                  | 60°C                           | 2 h/35 MPa                     | 40 mL/min                              | 2.42      | Lee et al. (2021)                     |
| Maceration and UAE | Brazilian rose pepper ( <i>Schinus terebinthifolius</i> Raddi) leaves | Ethanol/room temperature/600 W | 0.5 h                          | –                                      | 4.05      | Uliana et al. (2016)                  |
| UAE                | Nutmeg oleoresin  | Ethanol/22°C/120 W             | 10 min                         | –                                      | 6.03      | Morsy (2016)                          |
|                    | Myrtle extract  | Water /50°C/750 W              | –                              | –                                      | 24.58     | Asik, Atbakan                         |
|                    | Myrtle extract powder   |                                |                                |  | 22.55     | Kalkan, and Topuz (2021)              |

Allaboun, and Almasri 2019; Asik, Atbakan Kalkan, and Topuz 2021; Barla Demirköz et al. 2018; Bittencourt et al. 2019; Lee et al. 2021; Li et al. 2019; Sun et al. 2017; Uliana et al. 2016; Zermane et al. 2016).

In a study to extract volatile compounds from two species of *Hypericum* plant specimens using solid-phase microextraction (SPME) fibers, after mixing the plant powder in distilled water in a vial, the specimen was then placed in a sonicator for 15 min at 50°C. After the SPME fiber retainer was closed at the top of the vial, extraction operations were performed for the vial and the fiber for 40 min, and finally, the fiber was removed from the fiber retaining needle and inserted in the injection port of the GC-MS system. Compared to the HD method, this method showed a higher percentage of  $\alpha$ -pinene extraction for both species, so in the ultrasound-assisted headspace solid-phase microextraction (UA-HS-SPME) method for *Hypericum perforatum* and *Hypericum scabrum*, the rate of  $\alpha$ -pinene extraction was 15.2% and 48.4%, respectively. HD method reported 10.74% and 32.2%  $\alpha$ -pinene extraction rates (Ghiasvand et al. 2016).

#### Acidic and enzymatic pretreatment via distillation and UAE

In one study on mountain tea (*Sideritis* spp.) volatiles, the use of EAE before the UAE achieved better results than UAE alone. The total amount of identified compounds increased by 65% and yield by 25% compared to classic UAE. The enzyme was less sensitive to acid. Pretreatment with enzymes could improve the efficiency of distillation with water and UAE, but this effect certainly applies to acid pretreatment as well. As a result, incubation of plant material before distillation with water or UAE in citrate buffer, pH 4.8, significantly increases the extraction efficiency and the number of components obtained. Acid pretreatment was

also used to analyze cultivated *Sideritis raeseri* ssp *raeseri*. The amount of volatiles and yield in the EO obtained by acid pretreatment before HD increased by about 27–22%, respectively.  $\alpha$ -Pinene was one of the four main identified components in the plant. The amount of  $\alpha$ -pinene was generally examined in other methods. The highest amount of  $\alpha$ -pinene identified was related to acid pretreatment methods before UAE, acid pretreatment method before HD, EAE before UAE, UAE, HD method, maceration, and EAE before HD, reported at 15.4, 15.3, 14.2, 9.9, 7.2, 5.6, and 5.3 mg/100 g (Dimaki, Iatrou, and Lamari 2017).

#### Steam explosion method

One of the new and effective methods for extracting effective compounds in citrus EO is using steam explosion at high pressure and temperature. In this method, the extraction time is immensely shortened. In this process, the material is exposed to high-pressure saturated steam, which reduces the pressure on the vacuum tank through an angle valve. According to EO extraction from orange peels, it should be noted that in comparison with this method, the HD method was most used and the amount of  $\alpha$ -pinene in HD, and steam explosion methods were reported to be 1.2% and 0.72%, respectively (Golmohammadi et al. 2018).

#### Cold centrifugation method

Cold centrifuges are used in procedures where measuring antioxidant levels and preserving phenolic compounds are essential. As mentioned above,  $\alpha$ -pinene is also one of the compounds that have antioxidant properties (George et al. 2015). In one study, the EO was extracted from *Shiikuwasha* pulp using a fluorinated ethylene-propylene tube by cold centrifugation for 20 min at 4°C. The clock was compared

with a Clevenger device. Although the extraction efficiency by SD method was 6.99% > cold centrifugation (by 3.64%), the amount of  $\alpha$ -pinene in cold centrifugation and SD was 2.1 and 1.6%, respectively, indicating that the  $\alpha$ -pinene extraction in cold centrifugation is better than SD (Asikin et al. 2016).

### Microwave-assisted extraction (MAE)

MAE has been recognized as a method with several advantages over other methods, such as reduction of extraction time, costs, CO<sub>2</sub> emissions, and energy consumption (Cardoso-Ugarte et al. 2013). The chemical composition of *Cladanthus mixtus* EOs (CMEO) by MAE was determined through GC-MS. CMEO chemical analysis identified 44 constituents.  $\alpha$ -Pinene (5.7%), germacrene D (8.9%), and santolina alcohol (40.7%) were the major components (Zeroual et al. 2021).

Microwave-assisted hydrodistillation (MAHD) combines rapid heating in the microwave field with the traditional SE. This significantly enables saving of time, so the extraction can be completed in the meter of min (Moradi, Fazlali, and Hamed 2018). In one study, HD and MAHD methods were used to extract EO from mace (*Myristicae arillus*). The results exhibited that the MAHD method is more efficient than HD, and, in fact, MAHD required less energy (756 kJ) compared to HD (1095 kJ). The higher the power used, the higher the efficiency of the EO. Also, in the HD method, 7.03% of the EO was produced in 73 min, but in the MAHD process, the extraction efficiency was 8.6% in 42 min. In the MAHD method, the amount of the extracted  $\alpha$ -pinene was 18.4% at 300 W, 24.0% at 600 W, and 25.4% at 800 W while in the HD method, the amount of extracted  $\alpha$ -pinene was 22.2% (Megawati, et al., 2019). A similar study was performed on the extraction of EOs on *Ferulago angulata* (Apiaceae). In the MAHD method, the power of 0-1000 W was used, and the rate of  $\alpha$ -pinene extraction in this method was 13.9% and, for HD, was 18.2%. Also, the results revealed that the MAHD of *F. angulata* produced the highest EO yield (6.5%) as compared with EO prepared with the HD (2.65%) (Mollaei et al. 2019).

Solvent-free microwave extraction (SFME) is a hybrid method that is performed using dry distillation and microwave heating without adding any water or solvent at atmospheric pressure. Finally, the volatile compounds are concentrated and separated in one step (Lucchesi, Chemat, and Smadja 2004). To conduct the SFME technique, the aerial parts of *Sclerorhachis platy-rachis* (Boiss.) Podlech ex Rech.f. were heated in the microwave oven using an optimized fixed power of 800 W for 30 min. The chemical compositions of the isolated EOs were then determined by GC-MS, and the amount of  $\alpha$ -pinene extracted in this method was reported to be 12% (Nekoei and Mohammadhosseini 2018). The EO of *Limnophila aromatic* (Lam.) Merr aerial parts were isolated by SFME (Yingngam et al. 2021). The most influential variable affecting oil recovery was the irradiation time and the microwave power, respectively. EO yields varied from 0.20% to 0.24% (v/w)

depending on the season. A total of 47 compounds was found in the EOs, including perillaldehyde, limonene,  $\alpha$ -pinene, (Z)-4-caranone, and (E)-4-caranone. Hydrocarbons monoterpenes were the predominant compounds in oils.

### Green and solvent-free ultrasonic-microwave assisted extraction (UMAE)

In the separation of black and white pepper EO by the UMAE method, a schematic diagram of the UMAE device is shown in Figure 2c (Wang et al. 2018). The delivered power was 800 W of microwave input energy by the generator, while the energy of the input ultrasonic waves was applied by the ultrasonic converter with a constant power of 50 W/40 kHz, and they were used simultaneously to extract the EO from the pepper. The extraction temperature was 100 °C. The microwave and ultrasonic powers were 500 and 50 W, and the extraction time was 7 min. This method, compared to MAE and UAE, was much more efficacious because the higher efficiency of extraction and separation of effective compounds in this method was achieved. In addition, UMAE has been named the green extraction method due to the maximum extraction efficiency obtained, no solvent consumption and the shortest extraction time.  $\alpha$ -Pinene extraction rate for black and white pepper was 8.6% and 5.2%, respectively, which was > other methods (Wang et al. 2018).

### EAE combined with ultrasonic-microwave-assisted surfactant

In this method, the cell walls of the plant are split to release the intracellular chemical compounds and extract them with solvent, and this method is carried out to extract the maximum desired compounds from the plant. For this purpose, in the extraction of effective EO compounds from *Baeckea frutescens*, enzyme-based aqueous solutions were used as a pretreatment system. Also, the concentration of cellulose and hemicellulose was 10% (w/v), the concentration of Triton X-114 was 2% (w/v), the extraction time was 1.5 h, ultrasonic power was 478, and 535 W was selected as the microwave power. Combined analysis showed that  $\alpha$ -pinene (16.1%) was one of the most prominent compounds (Wan et al. 2021).

### Simultaneous ultrasonic/MAHD extraction (UMHE)

In a study on *Citrus medica* L. var. *sarcodactylis*, UMHE was first used to extract the EO, and the optimal conditions for the experiment were as follows: solvent to solid ratio 5.2:1 (mL/g), time extraction, and microwave power was 15.5 min and 816 W, respectively, as shown in Figure 2d (Zhang et al. 2019). Compared to the HD method, which had 1.5 h extraction time, the extraction time in this method was very short and appropriate, and the efficiency of this method was acceptable. In this study, the amount of  $\alpha$ -pinene extraction in UMHE and HD methods and extraction

through distillation with water by microwave (MHE) and SE were reported to be 3.0%, 2.6%, 2.7%, and 1.5%, respectively (Zhang et al. 2019).

### **Ohmic-assisted hydrodistillation (OAHD)**

OAHD is an advanced method for extracting EOs that uses a progressive ohmic heating process to perform distillation with water in a refined manner (Figure 2e; (Gavahian et al. 2012)). This method is used as a green method because less energy is needed per mL of extracted EO. This technology does not require any organic solvents for extraction and consumes much less energy to operate. One of the advantages of ohmic heating in EO extraction is that it causes rapid rupture of EO glands and their surrounding area, resulting in a shorter extraction process. The effect of NaCl is negligible compared to other parameters in this method, and NaCl shows a positive impact on reducing the initial temperature as well as shortening the onset time of oil accumulation during the work (Gavahian et al. 2012). In a study on *Vitex pseudonegundo*, the effects of different input voltages (100, 200, and 300 V) and NaCl concentrations (1%, 2%, and 3%) on EO extraction were investigated. The results showed that salt concentration and voltage were critical factors in the extraction that their regulation was directly related to the quality, and the amount of  $\alpha$ -pinene for voltages and different percentages of NaCl was reported as 25.3–30.2%. The addition of NaCl, also, significantly shortened the onset time of EO accumulation and reduced the required temperature for the extraction (Hashemi et al. 2019).

### **Hybrid solar distillation system**

In a study to extract EOs and active compounds on three plants, Eucalyptus, Peppermint and Pinus, an innovative system was used that consisted of a distillation apparatus, a primary reflector (Schaffler concentrator), condenser, steam receiver, and a fluorescent flask. An auxiliary biomass system is coupled with the distillation unit to equip the system effectively in case of seasonal climatic conditions or adverse weather conditions. Less than half of the year is estimated as the payback period for this distillation system. Extracted  $\alpha$ -pinene was reported to be 7.1% for Eucalyptus and 70.9% for Pinus. Overall, it can be concluded that solar-based distillation has proven to be a cost-effective process for extracting more EOs from medicinal plants (Afzal et al. 2017). The cost-effectiveness of solar-based distillation system for the extraction of EOs from medicinal plants is an advantage of this method.

### **Gas–liquid microextraction (GLME)**

GLME is developed from gas purge microsyringe extraction (Gp-MSE) (Zhao et al. 2020). Compared to GP-MSE, GLME substituted a 150 L receiving vial for the microsyringe. The sample is heated to evaporate target compounds from the sample matrix and then transferred using an inert gas carrier

to be collected in the receiving vial. GLME is a robust and sensitive extraction procedure for analyzing plant semiochemicals (Zhao et al. 2020). *Thuja koraiensis* Nakai was used as a representative plant to assess recovery, reproducibility, and peak number. The GC/MS analysis revealed  $\alpha$ -pinene,  $\beta$ -caryophyllene, linalool, totarol, camphene,  $\alpha$ -terpinenol, and  $\alpha$ -caryophyllene with concentrations (SD,  $n=3$ ) of 0.65 (0.06), 4.12 (0.15), 0.62 (0.05), 0.99 (0.08), 21.91 (0.25), 1.11 (0.07), and 0.063 (0.04)  $\mu\text{g g}^{-1}$ , respectively.

### **Needle trap microextraction (NTME)**

In various matrices, NTME has been introduced as a quick and straightforward extraction/isolation approach for VOCs (Figueira et al. 2020; Porto-Figueira, Pereira, Miekisch, et al. 2018; Porto-Figueira, Pereira, and Câmara 2018; Trefz et al. 2013). Because the sorbent particles are enclosed inside a needle trap device (Figure 2f; Figueira et al. 2020), NTME is mechanically more resistant than SPME. Furthermore, NTME is an exhaustive extraction approach, meaning that all the VOCs in the sample can be extracted fully, at least until the sorbent bed saturation (breakthrough) occurs. In addition, increasing the sample volume improves NTME sensitivity, and increasing the amount of the packed sorbent in needle trap device expands its capacity (Barreira et al. 2016; Kędziora and Wasiak 2017; Mieth et al. 2009). NTME, in conjunction with GC-MS analysis and chemometric processing, was applied to generate comprehensive volatile fingerprints for authentication purposes (Figueira et al. 2020). This method allows lemon peels' volatile composition (exocarp) (*Eureka* variety). In total, 75 VOCs were found, divided into four chemical groups: sesquiterpenes, monoterpenes, alcohols, and carbonyl compounds.  $\alpha$ -pinene, d-limonene, sabinene,  $\beta$ -pinene,  $\gamma$ -terpinene, and  $\beta$ -myrcene constituted over 50% of the volatile compound in various lemon varieties.

### **Indirectly suspended droplet microextraction (ISDME)**

ISDME combines directly suspended droplet microextraction (DSDME) and salting-out extraction of water-miscible organic solvents. ISDME was used to extract volatile compounds ( $\alpha$ -pinene, borneol, linalool, limonene, 2-phenylethanol, decanoic acid, and thymol) in fruit juice and rose-water (Babae and Daneshfar 2016). The sample solution was mixed with 2-propanol, and instantly, a homogeneous solution was created. The solution was agitated using a magnetic stirrer to create a constant vortex. 2-Propanol was separated and collected at the bottom of the continuous vortex by adding ammonium sulfate to the homogenous solution. ISDME provides several advantages over salting-out extraction and traditional DSDME, including: (i) it is faster than traditional DSDME; and (ii) sampling of water-miscible organic solvents is simple. Also, ISDME is a quick and straightforward extraction operation that saves energy and minimizes the cost analysis. Furthermore, compared to the most approaches, this method offers a

more comprehensive dynamic range, higher detection limit, and a faster extraction time, making it a viable tool for routine analysis.

### Electromagnetic induction heating (EMIH)-assisted extraction

In one study, berries and branches were exposed to this extraction method. The system was equipped with a pressure cooker placed on the induction plate (1800 W), the extraction temperature was 140°C, and the extraction time was 55 min. Extraction was performed under magnetic conditions, and the EO was collected by evacuation method and dried on anhydrous sodium sulfate and compared with extraction through the HD method, which was carried out with Clevenger for 1.5 h. For the extraction of  $\alpha$ -pinene from the EOs of berries and branches in the EMIH-assisted extraction method, the values of 44.8% and 67.7% were reported, while for the HD method, these values were 40.3% and 50.5%, respectively (Harhour et al. 2018). One of the advantages of this method is a significant increase in the extraction rate of polar compounds, for example phenolic compounds. Saving time is another benefit of this method. Compared to the conventional heating method, EMIH hand over rapid heating through the plant by producing heat even within the material to be heated. Hence, energy is directly reproduced in the core of the plant material, which provides consistency and homogeneity in heating (Megateli and Krea 2018).

### Purification and trapping (P&T) method

In P&T technique (Figure 3), an inert gas is bubbled via the sample, and the volatile analytes are transferred to an adsorbent trap. The adsorptive material is usually Tenax®, a synthetic polymer that does not react with the analytes but efficiently binds them under ambient conditions and releases them at an elevated temperature without chemical

modification. The trap is heated, and the volatiles are released or desorbed and transferred to GC. Finally, the trap is heated to a higher temperature than was used during the desorption step to remove residual analytes and moisture (Sparkman, Penton, and Kitson 2011). This method could be potentially applied for the extraction of  $\alpha$ -pinene although it has not been employed yet.

### Purge and trap (P&T) GC-MS

This technique could be applied for both liquid and solid samples, and is especially useful for volatile compounds since it needs just small level of samples, and could be run without organic solvents (Lee et al., 2015). P&T analysis is a technique for high-sensitivity detection of VOCs in wastewater, water, sludge, and soil with very low carryover. The extraction and identification of distinctive semi-volatile and volatile components of five monofloral honey varieties were performed via a P&T GC-MS system (Tananaki et al. 2022). A total of 124 compounds were identified. Octane,  $\alpha$ -pinene, and nonanal seem to characterize honeydew honey. The utilization of volatile profiles could lead to differentiation among the different monofloral honey kinds, according to multivariate statistical analysis of purge and trap GC-MS data.

In one study, finger citron samples were purged with nitrogen gas for 4 h after treatment with 4-methyl-2-pentanone and trapped into thermal desorption (TD) tubes filled with Tenax TA adsorbents. The TD tubes were then loaded into TDS3, and trapped volatiles desorbed at 280°C for 5 min, then cooled to -80°C with liquid nitrogen. The volatiles were injected (outlet split flow of 100 mL/min) into a capillary column via a valve and transfer line temperature of 280°C. 44 and 45 odorous compounds, including  $\alpha$ -pinene, were detected from flesh and peel of Jinghua Finger Citron by GC-MS, respectively. The results showed that P&T and HS-SPME could be used complementarily in odorous compound identification in Finger Citron (Song et al. 2018).

### Solid-phase microextraction (SPME) and headspace (HS)-SPME

SPME is an effective sample preparation method that combines several operations such as sample collection, extraction, enrichment of analytes, and separation from sample matrices, and uses solid, liquid, and gaseous samples to extract the analytes (Kataoka 2017). It is an adsorption-based sampling technique that uses this technique to adsorb analytes from a gas or liquid sample to a molten silica fiber through an adsorbent coating and, for a fixed time, fiber is part of the syringe needle. Important parameters in this method are extraction temperature, fiber type, equilibrium time, and sampling time. For example, in a study of *Aframomum danielli* seeds, temperatures of 20, 35, 50, and 65°C were examined by the HS-SPME method and results exhibited that the extraction efficiency of VOCs for  $\alpha$ - and  $\beta$ -pinene increased by 1.5 times at 50 and 65°C compared to 20 and 35°C. Also, in 40 min of extraction, compared to 10 min of

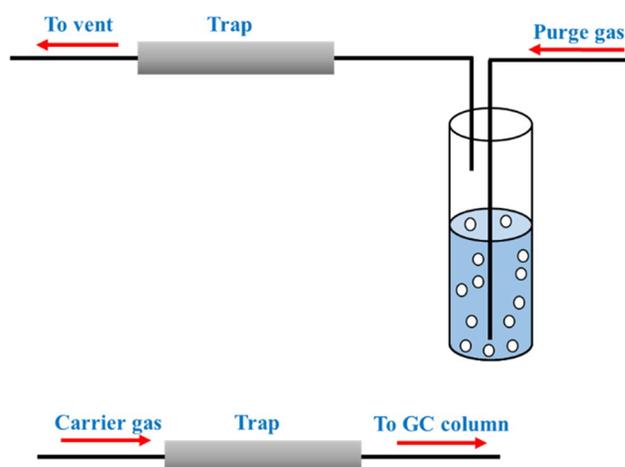


Figure 3. Purge gas bubbling via a sample containing volatiles directed to a trapping material; reproduced with permission from (Sparkman, Penton, and Kitson 2011).

extraction, about 1.25–1.75 more efficiency yield for  $\alpha$ -pinene was reported (George et al. 2018).

In a study, various factors were investigated to increase the extraction efficiency of volatile compounds and  $\alpha$ -pinene and to facilitate the transfer of volatiles. They concluded that equilibrium time (15 min), extraction temperature (32.5), and salt concentration (15%) are the most essential factors affecting steam pressure and the balance of flavoring compounds in the headspace (Mehta et al. 2018). In this extraction method, as in most extraction methods, factors such as plant type and growth conditions are influential. For example, in one study, it was reported that the rate of extraction of  $\alpha$ -pinene and other effective compounds in EOs in *Stachys cretica* and *Stachys lavandulifolia* was different in the pre-flowering, flowering, and post-flowering stages. For *Stachys cretica*, the amount of  $\alpha$ -pinene was 2.1%, 1.1%, and 1.2%, respectively, and for *Stachys lavandulifolia*, was 10.7%, 11.2%, and 10.9% before, during and after flowering, respectively. Therefore, for *Stachys cretica*, the amount of  $\alpha$ -pinene in the pre-flowering stage and for *Stachys lavandulifolia*, the amount of  $\alpha$ -pinene in the flowering stage was the highest (Sarıkaya 2018).

One of the SPME advantages is the possibility to sample directly the vapor phase in equilibrium with the matrix (HS-SPME), or the matrix extract or solution (liquid sampling-SPME), provided that suitable fibers are used (Cagliero et al. 2022). The purpose of this method is to determine and evaluate volatile compounds for EOs. In this regard, a research was conducted on two Brazilian medicinal plants called *Mikania laevigata* and *Mikania glomerata*, and in the SPME method of volatiles in the main space, fibers of Polyacrylate, polydimethylsiloxane/divinylbenzene (PDMS/DVB), and polydimethyl-siloxane (PDMS) were used. These

fibers were placed in a 20 mL vial to sample volatiles in liquid nitrogen, then the fibers were exposed to plant materials at 60°C for 5 min. Then, it was placed in gas chromatography. According to the results,  $\alpha$ -pinene for *M. laevigata* and *M. glomerata* was reported at 7.9% and 0.77% (Ueno and Sawaya 2019). In another study on leaves of *Rosa iberica* Stev., headspace examination was carried out by CombiPAL system with headspace auto-sampler, heater, and agitator, and  $\alpha$ -pinene was reported at 19.8% (Jowkar and Karami 2018).

In a study, HS-SPME-GC-MS was applied to extract EOs from leaves of *Juniperus chinensis* and *Juniperus seravschanica*. According to the results,  $\alpha$ -pinene for *J. chinensis* and *J. seravschanica* was reported at 27.2% and 33.1%, respectively (Khamis and Chai 2021). For a better understanding of the importance of the HS-SPME technique in the extraction of  $\alpha$ -pinene, some relevant studies are presented in Table 4 (Aguiar et al. 2019; Bazgir et al. 2020; Dadalı and Elmacı 2022; Karabagias et al. 2017; Ling et al. 2016; Mardani et al. 2016; Zouaoui et al. 2020).

#### Combined SPME and solvent-assisted flavor evaporation (SAFE)

The best method for the production of pure aromatic extracts, also without the loss of sensitive aromatic compounds or the formation of thermal artifacts produced during GC analysis, is the SAFE method (Zhu and Cadwallader 2019). One of the advantages of this method is the separation of volatiles from complex food matrices with high speed and accuracy. This method allows for separating volatiles from solvent extracts and foods that have continuous aqueous phase emulsions, such as milk or fruit juice (Engel, Bahr, and Schieberle 1999). In one study,

**Table 4.** Selected studies on the extraction of  $\alpha$ -pinene using the HS-SPME technique.

| Sample  | Solvent/T (°C)            | Time (min)<br>(extraction/equilibrium<br>or pre-conditioning/<br>sampling) | Fiber  | Yield (%)  | References                             |
|---|---------------------------|--|--|------------|--|
| <i>Scrophularia deserti</i>   | Water/60                  | 15/--  | PDMS   | 24.69      | Mardani et al. (2016)                  |
| Cold-pressed kernel oils from raw pistachio ( <i>Pistacia vera</i> L. Var Kerman)   | 1,2,3-Trichloropropane/50 | --/10/30   | Divinylbenzene/carboxen/polydimethylsiloxane | 0.4        | Ling et al. (2016)                     |
| Cold-pressed kernel oils from conventional roasted pistachio ( <i>Pistacia vera</i> L. Var Kerman)  |                           |  |  | 6.9        |  |
| Cold-pressed kernel oils from microwave roasted pistachio ( <i>Pistacia vera</i> L. Var Kerman)   |                           |  |  | 1.6        |  |
| Flower honeys from Samos Island   | Distilled water/45        | --/15/30   | Divinylbenzene/carboxen/polydimethylsiloxane | 25.74      | Karabagias,                            |
| Thyme honeys from Kos Island  |                           |  |  | 3.03       | Dimitriou, &                           |
| Fir honeys from Aitolokarnania  |                           |  |  | 16.97      | Halatsi, (2017)                        |
| Caramuri <i>Pouteria elegans</i> (A.DC.) Baehni   | 50                        | --/25/15   | Divinylbenzene/carboxen/polydimethylsiloxane | 21.77      | Aguiar et al. (2019)                   |
| <i>Crataegus pontica</i> K.Koch fruit   | 60                        | 20/--  | PDMS   | 15.40      | Bazgir, Ghaysouri, & Tahmasebi, (2020) |
| <i>Artemisia Campestris</i> L., <i>Artemisia herba-alba</i> Aso., <i>Juniperus phoenicea</i> L., <i>Rosmarinus officinalis</i> L., <i>Teucrium polium</i> L. and <i>Thymus algeriensis</i> L. | Water bath/70             | 15, 30, 45/20, 40, 60, 80/--   | Carboxen/polydimethylsiloxane                | 0.50–27.18 | Zouaoui et al. (2020)                  |
| <i>Mahaleb</i> seed kernels   | Cyclohexanone/30-70       | 10, 35, 60/0, 15, 30/--  | DVB/CAR/PDMS                                 | 0.24–5.10  | Dadalı and Elmacı (2022)               |

GC-olfactometry SPME and SAFE were used to analyze volatile compounds from cherimoya fruit. Eighteen deodorants were considered potentially active odor compounds, of which  $\alpha$ -pinene was one of the main compounds. Nonvolatile compounds with high vacuum distillation were removed using a SAFE apparatus, and after melting, the condensate was extracted three times with diethyl ether and dried over  $\text{Na}_2\text{SO}_4$ , and the amount of  $\alpha$ -pinene was reported to be 3.8% (Pino and Roncal 2016).

### Headspace stir-bar sorptive extraction

In this method, compared to other solvent-free methods, SPME in empty space mode has the highest extraction efficiency and provides the best sensitivity for target escapes provided that the samples are extracted at the right temperature and the continuous stirring time is performed correctly (Perestrelo, Nogueira, and Câmara 2009). In this method, a vial and a mixer strip covered with fiber and a mixer are used. In one study, fruit samples were placed in vials to extract volatile flavoring compounds, and then an EG (ethylene glycol) twisting silicone stirrer was used inside the space. To extract the volatile flavoring compound, vials containing an EG silicone stirrer were placed in a stirrer or heat block at 50 °C. Concentrations of the extracted  $\alpha$ -pinene from frozen omija, frozen-blended omija and freeze-dried samples omija were reported at 18.8, 30.3, and 273.3 mg/kg, respectively (Kim et al. 2019).

### Simultaneous distillation-extraction (SDE)

The SDE extraction technique combines SD for continuous extraction with a solvent or co-solvent mixture, resulting in faster extractions with lower extraction solvent volumes. *Rosmarinus officinalis* L is one of the plants in which  $\alpha$ -pinene is one of the main components. SDE was used to recover EOs from *Rosmarinus officinalis* L. (Ribeiro et al. 2021). Most of the chemicals examined had extraction efficiencies ranging from 40 to 70%, with precision ranging from 6 to 35%. The most prevalent chemicals in the EOs were  $\alpha$ -pinene, 1,8-cineole, (–)-bornyl acetate, (S)-(–)- $\alpha$ -terpineol, linalool, (–)-borneol, and 2,2,6-trimethylcyclohexanone (Ribeiro et al. 2021). This method proved to be a favorable way to get extracts free of chlorophylls and cuticular waxes. Assessing possible changes in the enantiomeric ratios of four different terpenes were performed during the growth of lemon fruit (Guadayol et al. 2018). The terpenes, including limonene, linalool,  $\alpha$ -pinene, and  $\beta$ -pinene, were extracted using SDE at reduced pressure (V-SDE) and SPME. The V-SDE method results showed appropriate monoterpenes extraction without affecting their enantiomeric distribution.

### Solvent extraction and solvent-assisted flavor evaporation

SAFE is considered the most effective method for preventing the loss of volatile compounds from aroma extracts.  $\alpha$ -Pinene is also a momentous component in the EOs of some herbs

and fruits, which is fragrant and volatile. The SAFE and solvent extraction of volatiles isolated from fruity, sweet, and slightly turpentine-like smelling *Spondias mombin* L. fruit pulp yielded 39 aroma-active constituents with flavor dilution factors between 4 and 1024 (Neiens, Geißlitz, and Steinhaus 2017). Aroma extract dilution analysis showed 33 previously reported compounds and eight compounds that had not been reported in *S. mombin* fruit before. The turpentine-like aroma was attributed to  $\alpha$ -pinene, (Z)- $\beta$ -ocimene, and myrcene. Static headspace analysis (SHDA) and SAFE, which are efficient in isolating highly volatile and semi-volatile compounds, respectively, were used to obtain fresh ginger's more comprehensive volatile profile (Pang et al. 2017). GC-olfactometry-MS was used to identify aroma-impact compounds, and aroma extract dilution analysis (AEDA) and SHDA were used to filter the most intense odorants further. According to SHDA,  $\alpha$ -pinene and eucalyptol were the most common headspace odorants. Also, three highly volatile components found by SHDA were not identified by AEDA. Whereas 34 low-volatility, high-polarity components were only detected by AEDA, indicating the complementary nature of AEDA and SHDA and the importance of using both approaches to evaluate ginger's scent accurately.

Also, headspace SPME and SAFE were used to extract volatiles from peel oil of an Huanglongbing-tolerant mandarin hybrid (Huang et al. 2017). AEDA identified the essential aroma components as  $\alpha$ -pinene,  $\beta$ -caryophyllene,  $\beta$ -myrcene, limonene, and linalool, each having a flavor dilution value  $\geq 128$ . Limonene was the most prevalent fragrance component quantitatively. Terpenes and aldehydes were the most common odorants found in the peel oil, which matched the earlier research. The compounds of  $\beta$ -myrcene,  $\alpha$ -pinene,  $\beta$ -caryophyllene, linalool, terpinolene, limonene, and decanal created aromas of the peel oil.

### Comparison of the efficiency of different extraction methods for $\alpha$ -pinene

Four different agro-industrial wastes were subjected to three different extraction procedures to determine their volatile terpene content (Bier et al. 2016). Various extraction techniques and solvents were more appropriate for each substrate, including pine wood shavings, pine needles, orange waste, and apple pomace. For apple pomace, the liquefied petroleum gas (LPG) approach yielded lower terpene concentrations than the Soxhlet method. Also, the dichloromethane: *n*-pentane extraction did not detect the three primary terpenoids. For the pine wood shavings, the LPG approach had satisfactory results for the oil composition but yielded the lowest extraction yield. Among the tested procedures, the LPG approach produced the best results for orange waste. This approach had the highest yield and the greatest limonene concentration. Among the examined residues, the orange waste had the highest concentration of terpenoids, so this substrate is the most typical for demonstrating that the precision of the LPG method for terpene extraction produced high concentrations and purity. The LPG process has a strong affinity for terpenoids, providing clean

extractions free of undesirable chemicals with waxy properties. Compared to procedures that use heat, this method resulted in less breakdown of the chemicals. As a result, this approach should be researched further for commercial applications with terpenes and other molecules with similar properties, such as EO constituents and aroma compounds. This method could be used as a phase in terpene recovery operations as an intermediary stage. Furthermore, this method is easily adaptable to laboratory extractions. For a more comprehensive overview of the extraction methods of  $\alpha$ -pinene, some relevant studies are summarized in Table 5.

## Purification of $\alpha$ -pinene

### Chromatographic techniques

Researchers tested the antibacterial activity of EO extracted from *Echinops kebericho* Mesfin Tuber (Deyno et al. 2020). The chromatographic technique was used for purification and isolation of components of EO, including  $\alpha$ -pinene, while spectroscopy was used for identification. Briefly, in a separatory funnel, the dried extract was soaked in distilled water before being added to an equal volume of hexane, agitated carefully, and allowed to stand until it separated into two layers. The hexane layer was separated three times, and the operation was repeated. The exact process was used to prepare ethyl acetate, chloroform, and *n*-butanol fractions.

Gradient solvent systems (various solvents from non-polar to highly polar) were designed and evaluated for the optimum separation of diverse organic components. The mobile phase was introduced slowly from the top of a column filled with silica gel. The sample was inserted in the top of the column once it was ready. A small layer of cotton separated the column from the incoming mobile phase, preventing the column from being disrupted by an influx of the mobile phase. After that, the mobile solvent was allowed to flow down the column. The ethyl acetate fraction's weighed dried residue was adsorbed to silica gel and separated on a column. The elution was carried out in the following order with different volume ratios: hexane: ethyl acetate, and a mixture of ethyl acetate: methanol. Thin-layer chromatography (TLC) was used to examine the fractions obtained. Preparative TLC was used to extract six EO fractions from *Eucalyptus saligna* leaf litter, which were then evaluated using GC-MS (Silva et al. 2020). Seedling growth and germination were evaluated using oil, its fractions, and key compounds ( $\alpha$ -pinene and/or 1,8-cineole) diluted in an aqueous solution. The key products of terpene oxidation in the atmosphere are organic nitrate esters. Using column chromatography, researchers purified nine nitrate esters derived from  $\alpha$ -pinene, (+)-3-carene, perillic alcohol, limonene, and  $\beta$ -pinene (McKnight et al. 2020). The availability of these components will allow for more in-depth research into the structure-reactivity connections of aerosol

**Table 5.** Selected studies on the comparison of extraction methods for  $\alpha$ -pinene from one fruit or plant.

| Sample  | Extraction   | $\alpha$ -Pinene yield (%)  | References                                   |
|---|--|---|--|
| Chinotto ( <i>Citrus myrtifolia</i> Raf.)   | HD   | 1.46  | Salvo et al. (2019)                          |
|   | SLE  | 0.34  |  |
|   | Hand-squeezing   | 0.94  |  |
|   | Blending + SLE   | 1.88  |  |
|   | Vegetable strainer + funnel (Fruits)   | 0.48  |  |
| <i>Thymus Pubescens</i>   | Solvent extraction (acetone and ethanol) and HD (Foral parts)  | 18.17 for acetone, 10.71 for ethanol, 0.9 for HD                              | Mohseni and Rad (2018)                       |
| Sawdust of pines  | Solvent-free microwave extraction (SFME) and HD  | 76.6 for SFME and 77.3 for HD   | Karaođul and Alma (2019)                     |
| <i>Laurus nobilis</i> L.  | SFME and HD (Leaves)   | 4.6 for SFME; 9.2 for HD  | Bendjersi et al. (2016)                      |
| <i>Rosmarinus officinalis</i> L.  | SFME (Leaves)  | 16.95%  | Al Zuhairi et al. (2020)                     |
| Bergamot juice  | Methanol/Acetone/Hexane (Fruits)   | 375 mg kg <sup>-1</sup>   | Multari et al. (2020)                        |
| Lemon juice   |  | 76.9  |  |
| Orange juice  |  | 39.0  |  |
| Mandarin juice  |  | 19.0  |  |
| Bergamot pomace   |  | 211   |  |
| Lemon pomace  |  | 95.7  |  |
| Orange pomace   |  | 11.1  |  |
| Mandarin pomace   |  | 188   |  |
| <i>Vitex agnus-castus</i>   | Ethanolic/aqueous extracts (Leaves)  | 15.2  | Keikha et al. (2018)                         |
| Lamiaceae ( <i>Stachys officinalis</i> (L.) Trevisan subsp. <i>officinalis</i> )            | Solvent extraction ( <i>n</i> -hexane) (Flower)  | 7.0   | Giuliani et al. (2017)                       |
| <i>Juniperus communis</i> L. and <i>Juniperus virginiana</i> L.                             | Ethyl acetate and tetrahydrofuran for refluxing extractions, ethyl acetate for sonication (Branches, berries, and needles) | 1.51–19.73 for refluxing and 26.70 for sonication                             | Plesa et al. (2017)                          |
| Iranian dill ( <i>Anethum graveolens</i> L.) and savory ( <i>Satureja sahendica</i> Bornm.) | Solvent-assisted flavor evaporation (SAFE) (Aerial parts)  | 4.49 $\mu\text{g g}^{-1}$ for dill and 267.08 $\mu\text{g g}^{-1}$ for savory | Amanpour, Kelebek, and Selli (2017)          |
| <i>Myrtus communis</i> L.   | Simultaneous distillation-extraction )SDE(   | 36.19   | Saaidpour and Jahannamaie (2019)             |
| <i>Juniperus oxycedrus</i> L. ssp. <i>oxycedrus</i> .                                       | SDE  | 30.1–66.4   | Llorens-Molina, Ygueravide, and Vacas (2019) |
| Cascarillo ( <i>Ocotea aff. O. caparrapi</i> )  | SDE (Leaves)   | 23.9  | Tafurt García and Muñoz Acevedo (2018)       |
| <i>Anemia tomentosa</i> var. <i>anthriscifolia</i>  | SDE (Leaves)   | 9.3–24.3  | Castilho et al. (2018)                       |

production and processing and specific investigations into organic nitrate esters' aqueous-phase interactions.

During fractionation, the active chemical compounds present in a solution can be identified and isolated to achieve a fraction or own isolation of the biologically active compound. The acaricidal effect was evaluated by the fractions, and EO derived from *Laurus nobilis* leaves on *Rhipicephalus (Boophilus)* (Fernandez et al. 2020). In the Larval Packet Test, FR8:  $\alpha$ -terpineol (79.2%) and FR1:  $\alpha$ -pinene (12.6%), 1,8-cineole (12.7%),  $\beta$ -pinene (13.5%), sabinene (37.8%) were identified as the compounds with the most significant larvicidal potential.

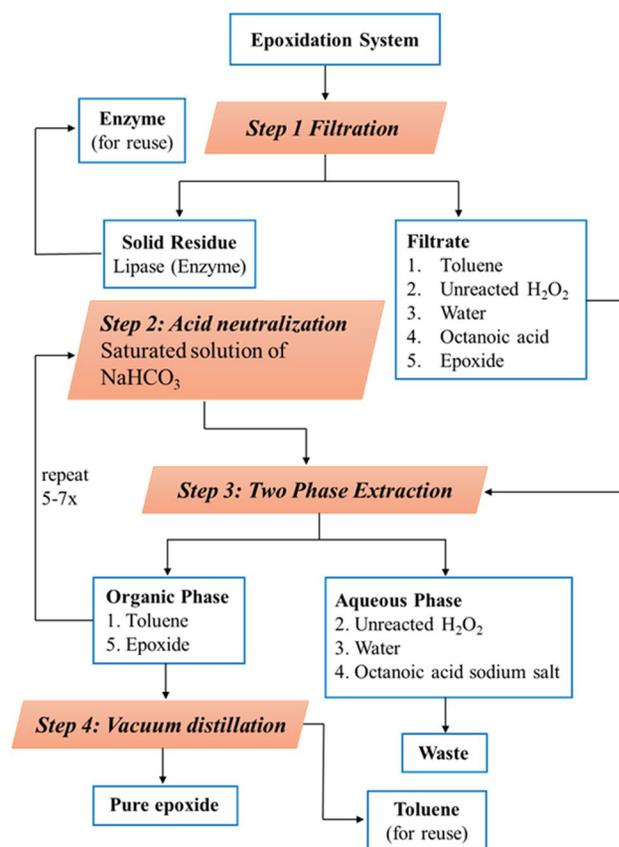
### Detoxification technique

For detoxification and production of isoprenoids with engineered *E. coli*, inhibitors generated after hydrolysis of microalgae biomass (with sulfuric acid) is removed first (Wang and Yang 2017). In order to remove the inhibitors, five steps are performed respectively (Wang and Yang 2017; (i) Adjusting the pH of the hydrolysate to pH = 10 with sodium hydroxide and then readjusting the pH to 5; (ii) Neutralization of the hydrolysate with calcium hydroxide; (iii) Regulation of the pH of the hydrolysate to 5 with sodium hydroxide; (iv) Addition of anion exchange resin to the hydrolysate; (v) Adjusting the pH of hydrolysate to 7 and then readjusting the pH to 5.5. The fermentation concentrations achieved in the above-mentioned study through this technique was as follows:  $\beta$ -pinene (17.4 mg L<sup>-1</sup>), isoprene (223.2 mg L<sup>-1</sup>), and  $\alpha$ -pinene (382.2 g L<sup>-1</sup>), using the detoxified hydrolysate as the carbon source.

An alcoholic extract of *Carum copticum* seeds and its isolated compounds were tested in an immunomodulatory evaluation on Swiss albino mice (Sonar, Singh, and Saraf 2016). After the ethanolic extract (100g) suspension in distilled water (200 mL) and extraction with chloroform, *n*-hexane, and *n*-butanol in a separatory funnel, all of the fractions were concentrated and then dried entirely in a desiccator at low pressure. The *n*-hexane fraction was eluted from silica gel column chromatography with ethyl acetate and *n*-hexane as eluents with increasing polarity. After monitoring their TLC, 10 mL fractions were obtained, and similar fractions were pooled. As a yellow-colored oily liquid, various compounds were produced from the eluent of *n*-hexane: ethyl acetate. This mixture was chromatographed over a silica gel column using methanol and chloroform as eluents with increasing polarity to get three oily liquids. This method was repeated to acquire enough separated oils for the pharmacological activity tests. Carvacrol, *p*-cymene, and  $\alpha$ -pinene were found in the *n*-hexane fraction. The crude extract's LD50 was 4500 mg kg<sup>-1</sup>, while the literature values for  $\alpha$ -pinene, carvacrol, and *p*-cymene were 3700, 810, and 4750 mg kg<sup>-1</sup>, respectively.

### Epoxidation system

The design of experiments was used to successfully optimize the process parameters of *Candida antarctica* lipase mediated



**Figure 4.** The epoxide purification process based on optimized lipase-mediated epoxidations; adapted with permission from (Ranganathan et al. 2016).

epoxidation of monoterpenes (Ranganathan et al. 2016). Purifying the epoxides led to an isolated yield of 62.5, 88.8, and 72.3 for limonene, 3-carene, and  $\alpha$ -pinene, respectively. Epoxides of terpenes undergo ring opening to create diols when exposed to a strong base, such as a 10 M NaOH solution. As a result, sodium bicarbonate (saturated quantities), as a weaker base, was utilized 5–7 times to completely neutralize the leftover acid concentration used in this method, namely the octanoic acid (C8) as a sodium salt. This salt could be used as a profitable by-product of the process if it is developed for industrial efficiency. Figure 4 depicts the specific process for performing this purification step.

### Conclusion

This work reviewed reports on  $\alpha$ -pinene extraction and purification methods in the last six years. Various extraction methods such as HD, SD, SFE, UAE, UA-HS-SPME, cold pressing method, steam explosion method, cold centrifuges method, MAHD, EMIH-assisted extraction, ohmic-assisted hydrodistillation, headspace stir-bar sorptive extraction, SFME, SDE, P&T GC-MS, VASE, SAFE, needle trap micro-extraction, and so on were investigated and discussed. Also, the purification of  $\alpha$ -pinene from components of EO was studied. The respective section mainly studied chromatographic methods as the most widely used  $\alpha$ -pinene purification methods. The results of this study are significant for

selecting a practical and effective method for the extraction and purification of  $\alpha$ -pinene. A common advantage of conventional methods such as the cold press method along with the VASE with the SFE method is the use of low extraction temperatures and the preservation of the  $\alpha$ -pinene nature. One of the significant advantages of new extraction methods compared to conventional methods is the reduction of time, cost, and energy. However, the application of some novel methods, such as UAE, in industry scale is still problematic because of their intricate design data. The LPG process has a strong affinity for terpenoids, providing clean extractions free of undesirable chemicals with waxy properties. Compared to procedures that use heat, this method resulted in less breakdown of the chemicals. As a result, this approach should be researched further for commercial applications with terpenes and other molecules with similar properties, such as EOs and aromatic compounds. This method could be used as an intermediary stage in terpene recovery operations. Furthermore, this method is easily adaptable to lab extractions. As a future research recommendation, more cost-effective “green” extraction methods with a faster extraction time and lower consumption of solvents could be explored although some initial steps have already been taken. In fact, the focus on eco-friendly extraction processes is essential as environmental pollution is regarded as a serious global issue these days.

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## Abbreviations

|         |  |
|---------|--|
| AEDA    | aroma extract dilution analysis              |
| CXE     | carbon dioxide expanded ethanol              |
| CMEQ    | <i>Cladanthus mixtus</i> EOs                 |
| CTV     | citrus species to the Tristeza virus         |
| DSDME   | directly suspended droplet microextraction   |
| EAE     | enzyme-assisted extraction                   |
| EMIH    | electromagnetic induction heating            |
| EOs     | essential oils                               |
| GC-MS   | gas chromatography-mass spectrometry         |
| GLME    | gas-liquid microextraction                   |
| Gp-MSE  | gas purge microsyringe extraction            |
| GC-FID  | GC with flame-ionization detection           |
| HS-SPME | headspace solid-phase microextraction        |
| HPLC    | high-pressure liquid chromatography          |
| HPTLC   | high-performance thin-layer chromatography   |
| HD      | hydrodistillation                            |
| ISDME   | indirectly suspended droplet microextraction |
| LLE     | liquid-liquid extraction                     |
| LPG     | Liquefied petroleum gas                      |
| MAE     | microwave-assisted extraction                |
| MAHD    | microwave-assisted hydrodistillation         |
| NTD     | needle trap device                           |
| NTME    | needle trap microextraction                  |

|                   |  |
|-------------------|--|
| OAHD              | ohmic-assisted hydrodistillation                           |
| PDMS              | polydimethylsiloxane                                       |
| PDMS/DVB          | polydimethylsiloxane/divinylbenzene                        |
| P&T               | purification and trapping                                  |
| ROS               | reactive oxygen species                                    |
| V-SDE             | SDE at reduced pressure                                    |
| SDE               | simultaneous distillation-extraction                       |
| SLE               | solid-liquid extraction                                    |
| SPME              | solid phase microextraction                                |
| SAFE              | solvent-assisted flavor evaporation                        |
| SE                | solvent extraction   |
| SFME              | solvent-free microwave extraction                          |
| SHDA              | static headspace analysis                                  |
| SD                | steam distillation   |
| scCO <sub>2</sub> | supercritical carbon dioxide                               |
| SFE               | supercritical fluid extraction                             |
| TD                | thermal desorption   |
| TLC               | thin-layer chromatography                                  |
| UAE               | ultrasound-assisted extraction                             |
| UA-HS-SPME        | ultrasound-assisted headspace solid-phase microextraction  |
| UMAE              | ultrasonic-microwave-assisted extraction                   |
| UMHE              | ultrasonic/microwave-assisted hydrodistillation extraction |
| VASE              | vacuum-assisted sorbent extraction                         |
| VOCs              | volatile organic compounds                                 |
| VTs               | volatile terpenes  |

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