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Effects of different drying methods on the volatile components of selected spices

MASTER'S THESIS

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Declaration

I hereby declare that I have done this thesis entitled **"Effect of different drying methods on the volatile components of selected spices"** independently; all texts in this thesis are original, and all the sources have been quoted and acknowledged by means of complete references and according to Citation rules of the FTA.

In Prague April, 2022

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Osho Dorcas Odunayo

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Abstract

This study was conducted to examine the effect of different drying methods on the volatile components of selected spices which include parsley, sage, basil, rosemary, ginger, and thyme. The volatiles were extracted with a Soxhlet extractor and subsequently analyzed using gas chromatography with mass detection. The result revealed that commercial parsley contained 76.48 % apiol while parsley dried at room temperature and 45 °C oven-drying contained 65.45 % and 93.64% apiol, respectively. A total of 23 volatile compounds were identified in rosemary. The analysis identified Eucalyptol as the most abundant volatile compound in commercial rosemary (47.73 %), air-dried rosemary (22.95 %) and oven-dried rosemary (19.28 %). A total of 27 volatile compounds were identified in sage. The analysis identified 2-Bornanone, Eucalyptol and Epimanool as abundant volatile compounds in sage. This study identifies a total of 32 volatile compounds in ginger. The analysis identified Curcumene, β-Bisabolene, and β-Sesquiphellandrene as abundant volatile compounds in commercial and air-dried ginger. The study found β-Elemene, epi-β-Caryophyllene, and alfa-Copaene as abundant volatile compounds in oven-dried ginger. This study identifies a total of 31 volatile compounds in basil. The result identified Eugenol, α-Bergamotene, and Linalool as abundant volatile compounds in commercial, air-dried, and oven-dried basil. A total of thirteen compounds were identified and Thymol, γ-Terpinene and m-Cymene were identified as the abundant compounds in thyme. Commercial thyme contained 71.01 % Thymol while thyme dried at room temperature and 45 °C oven-drying contained 58.70 % and 59.98 % Thymol respectively. The study concludes that oven-drying at 45 °C is ideal for drying ginger, basil and thyme as it increased the availability of some volatile compounds, while drying at room temperature is ideal for drying parsley as there are more volatile compounds in the essential oil.

Keywords: drying, spices, essential oil, volatile compounds, GC/MS, oven-drying, air-drying, parsley, sage, rosemary, ginger, basil, thyme.

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List of abbreviations

Comm – Commercial

EO – Essential oil

GC/MS – Gas chromatography/Mass spectrometry

GH – Greenhouse

ISO – International Organization for Standardization

Lit – Literature

LC/MS – Liquid Chromatography/Mass Spectroscopy

MAE - Microwave-Assisted Extraction

Min – Minutes

NIST – National Institute of Standards and Technology

Obs – Observed

OS – Open Sun

PEF – Pulsed Electric Field

PLE - Pressurized Liquid Extraction

PUB – Published

R temp – Room Temperature

RH – Relative Humidity

RF– Radio Frequency

RT – Retention Time

SFE – Supercritical Fluid Extraction

$SCCO_2$ – Supercritical CO_2

UAE - Ultrasound-Assisted Extraction

1. Introduction and Literature Review

1.1 Introduction

A spice is a plant or herb's dried seed, fruit, root, bark, or flower that is used in tiny quantities for flavour, colour, or as a preservative (Sachan et al. 2018). The International Organization for Standardization defines spices and condiments as "natural plant or vegetable products or mixtures thereof in whole or ground form that are used for adding flavour, smell, and piquancy to and flavouring meals" (Padakatti & Meti 2020). Spices have an important role in the diet as flavouring, colouring, or preservation agents, and they are utilised all around the globe (Arvind et al. 2016). Spices are essential components of our daily diet, even if we only take a little quantity of them (Khanum et al. 2001). Spices have long played an important part in both ancient and contemporary culinary preparations (Balasasirekha 2014).

The bulk of the key components of spice ingredients are carbohydrates, protein, and trace minerals. Tannins, resins, pigments, volatile, essential, and fixed oils are present in trace levels and account for just a tiny part of the dry matter (Ugwuona 2014). Commercial spices include red pepper, onions, sage, ginger, nutmeg, clove, cinnamon, mustard, curry, turmeric, rosemary, and garlic. Spices provide flavour, savour, and pungency to meals. The majority of spices are fragrant, aromatic, and pleasurable. Spices in food can offer additional advantages such as lowering salt and sugar levels, preventing spoilage, and improving texture (Ravindran et al. 2002).

Spices are known to fight cancer and a variety of heart diseases owing to the phytochemicals they contain. Spices are employed as culinary components as well as in a range of pharmaceuticals, fragrances, cosmetics, and natural colours. Many spices have been used in traditional medicine for a long time. Many regional cuisines are well-known for their reliance on exotic spices. Turmeric is utilised in Italian food, basil, garlic, and oregano in Italian and Greek cuisine, and lemon grass, ginger, and chilli peppers in Thai cuisine (Satia-Abouta et al. 2002). Meanwhile, since spices have a weak internal structure and are very perishable, an efficient technique of preservation is required to lengthen shelf life and increase value (Majumder et al. 2021).

Drying is a time-honoured and unrivalled physical technique of food preservation that is used for both direct manufacture of food products and extra processing in the food industry. It has historically been a valuable and widely used conservation practise, ensuring a steady supply of food and medical supplies throughout the year. Drying used to be a simple and natural process since it was driven by the sun. It has grown more sophisticated and complicated in recent years as a result of the use of a huge quantity of equipment and the careful study and optimization of drying parameters at each step of the process. Emerging innovative approaches have been intensively investigated in terms of chemical and metabolic changes in the product throughout the dehydration process. Drying not only preserves the product but may also increase material quality, as in spices, medicinal plants, herbs, bioactive enzymes, and nuts, which may create value-added substances during drying (Szychowski et al. 2018; Rahman 2020).

Drying is universally acknowledged as the best technique for preserving fruits, vegetables, and herbs since it reduces their volume and weight, saving packing, storage, and transportation costs. Furthermore, flavour and texture characteristics are adjusted, resulting in a new generation of things such as snacks that may be a healthy alternative to other commercial products such as sweets (Sehrawat et al. 2018). Furthermore, eliminating water reduces microbe development and harmful chemical reactions, resulting in a longer storage duration (Amit et al. 2017). When the water activity is less than 3 %, this is commonly performed. Thus, there are various types of drying methods based on the technique of removing water, such as thermal drying, which is further subdivided into air-drying, low air environment drying, and modified atmosphere drying; osmotic dehydration, which uses a solution to remove the water; and mechanical dewatering, which uses physical force to dry (Rahman 2020).

Traditional thermal and non-thermal drying processes are used to dry spices. Because of its simplicity and low cost, direct solar or open sun drying (OS) has been the most popular in tropical areas from ancient times. However, it is weather dependant, the product is prone to contamination, and drying parameters are sometimes difficult to control. In order to overcome the limits of OS, indirect sun drying and greenhouse drying (GH) may be used. In

addition to solar energy, biomass and geothermal energy are used as heat sources to power the drying medium (Ananno et al. 2020).

Environmental conditions have an impact on the volatile components of spices, particularly EOs (humidity, temperature, and velocity). The drying temperature is the most important factor in keeping the active components of volatile oil in gland cells, which are very sensitive to temperature rises. After harvest, the surface area of gland hairs declines in lockstep with cell death, possibly leading in considerable water loss. Drying accelerates water loss from the whole tissue, including gland hairs (Karami et al. 2017). In general, high temperatures alter the quantity and quality of essential oils in spices not only during drying, but also during storage (Fonseca et al. 2020).

Because drying decreases the weight and volume of the plant, which benefits transport and storage, it may also assist to ensure a steady supply and facilitate the marketing of spices (Majumder et al. 2021). As a consequence, appropriate drying techniques are necessary, including the use of temperature and humidity values for drying air to permit a speedy decrease in moisture content without affecting the quality of the active compounds in the spices.

1.2 Spices

A spice is a dried seed, fruit, root, bark, or vegetative component used as a flavouring and sometimes as a preservative by killing or slowing the development of harmful germs in tiny doses (Gobie 2019). Many of these substances are also found in medicine, religious rites, cosmetics, fragrances, and vegetables. Turmeric, for example, is used as a preservative; Licorice is used as a pharmaceutical; and garlic is utilised as a vegetable (Dalby 2001).

Since antiquity, spices have been used as preservatives, colourants, and flavour enhancers. Spices, which have long been used as a foundation for traditional medicine in many nations, have also been the subject of study, particularly by the chemical, pharmaceutical, and food industries, owing to their potential for health benefits. In vitro and in vivo studies have shown that these chemicals serve as antioxidants, digestive stimulants, hypolipidemic agents, as well as having antibacterial, anti-inflammatory, antiviral, and anticancerogenic activities. These

beneficial physiological effects may have significant preventative implications in a variety of diseases (Viuda-Martos et al. 2011).

Spices and herbs have been used as preservatives, colourants, flavour enhancers, and medicinal substances for thousands of years. With a 20-30 % growth, the use of spices and herbs as flavouring agents in food has become a major trend throughout the globe. Spice and herb trends vary around the globe, based on regional cuisines. Traditional spice and herb-based dishes abound. Turmeric, for example, is used in Indian cuisine, while basil, garlic, and oregano are used in Italian and Greek cuisines, and lemongrass, ginger, cilantro, and chilli peppers are used in Thai cuisine, indicating the global use of spices and herbs. According to studies, the most often used spices in 36 countries throughout the globe were onion, garlic, ginger, and peppers (Kaefer & Milner 2008).

Several spices and active compounds, including ginger, garlic, fenugreek, onion, curcumin, capsaicin, lemongrass, and cinnamon, have been linked to lower cholesterol (Srinivasan 2006). Garlic may help reduce blood pressure (Rahman 2007). According to certain studies, curcumin may be able to prevent strokes and reduce vascular edema in those who have had a hemorrhagic stroke (Singletary 2020). Spices and herbs have been demonstrated to help diabetics by slowing stomach emptying, enhancing insulin responsiveness, and strengthening antioxidant defences. Cumin seeds, ginger, mustard, curry leaves, coriander, curcumin, and fenugreek were shown to cause low blood sugar levels (Kochhar 2008; Singletary 2020). The use of cinnamon as a stimulant for the treatment of type 2 diabetes is very promising, but as with other spices, well-controlled clinical studies should be carried out before making conclusive pronouncements (Gruenwald et al. 2010).

An alcoholic extract of fenugreek seeds has been shown in studies to improve blood glucose levels, body weight, and the development of cataracts in the elderly. Fenugreek, garlic, ginger, and red pepper have all been linked to lower cholesterol levels. Curcumin, fenugreek, capsaicin (chilli), and ginger all aid in the synthesis of bile acid. Many other medicinal herbs lack antioxidants and phenolics, but oregano and rosemary mint do (Patel et al. 2011). Oregano contains significant amounts of rosmarinic acid, protochatechuic acid, quercetin, pcoumaric acid, and protein (McCue et al. 2004). Saffron has antioxidant, anti-inflammatory,

antihypertensive, and calming properties, as well as cardiovascular, lipid, insulin resistance, and tissue oxygenation advantages (Srivastava et al. 2010; Kianbakht & Hajiaghaee 2011).

1.2.1 Parsley

Parsley (*Petroselinum crispum* Mill.) is a common spice in the Mediterranean and Middle East countries, where it is used in a variety of meals and culinary preparations. Other known historic applications of parsley as a medicinal and culinary plant include its usage to flavour Chinese, Mexican, South American, Indian, and Southeast Asian cuisines (Farouk et al. 2017). Parsley, a well-known source of fragrant leaves and roots, is also used as a raw material in the synthesis of resinoids, oleoresins, and lipids such as palmitic, oleic, linoleic, and petroselinic acids (Snoussi et al. 2016).

Parsley leaves are used as condiments, garnishes, and flavouring elements in fresh, dried, and dehydrated forms. The leaves and seeds of parsley may be used to extract the essential oil, which is used as a flavouring or aroma in perfumes, soaps, and lotions (Farouk et al. 2017). The commercial essential oils of parsley are produced mostly from the seeds or plants picked before maturing during seed development (Petropoulos et al. 2004). Aziz et al. (2013) and Sabry et al. (2013) investigated Egyptian hydrodistillate oils of parsley and discovered that phenylpropanoids and terpenes were the main ingredients.

The chemical composition of parsley varies depending on a number of circumstances, therefore it differs not only across various sections and varieties of the plant but also between different samples of the same portions of one variety (Punoševac et al. 2021). The most prominent parsley chemicals include myristicin, apiol, 1-allyl-2,3,4,5-tetramethoxybenzene, -phellandrene, 1,3,8-p-menthatriene, -pinene, terpinolene, apiin, oxypeucedanin, and falcarinol. Parsley has a long history of use in the treatment of urinary tract disorders, and modern in vitro and in vivo studies reveal numerous effects of various parsley preparations such as diuretic, antiurolithiasis, hypouricemic, hypolipidemic, hypoglycemic, hypotensive, antioxidant, anti-inflammatory, and antiplatelet effect (Punoševac et al. 2021).

1.2.2 Sage

Sage (*Salvia officinalis* L.) is an essential oil plant that is also used as a medicinal herb and spice. Sage has a broad spectrum of biological actions, including antibacterial, fungistatic,

astringent, antiseptic, antifungal, and digestive properties (Baj et al. 2013). Sage leaves are employed in antibacterial and astringent herbal mixes, Septosan and Dentosept, whilst essential oil distillated from sage may be beneficial in aromatherapy (massage, bath, inhalation), as well as bacterial infections, cough, and bronchitis (Ali et al. 2015). Some of the essential oil's constituents are microbiologically active (Ramadan et al., 2019). Daferera et al. (2003) reported that sage inhibited the growth of *Botrytis cinerea*, *Fusarium* sp., and *Clavibacter michiganensis* microorganisms.

According to Bruneton (1999), commercial sage oil is characterised by thujones, with -thujone typically predominating (18–43 % over -thujone (3–8.5 %), camphor (4.5–24.5 %), 1,8-cineole (5.5–13 %), humulene (0–12 %), pinene (1–6.5 %), camphene (1.5–7 %), and bornyl acetate (2.5 % maximum). The oil contains high levels of -thujone, which was assumed to be the hallucinogenic component of absinthe and the source of absinthism. This, however, has been shown to be untrue (Lachenmeier et al. 2006). Nonetheless, excessive dosages of thujone elicit convulsions by inhibiting GABA-gated chloride channels (Höld et al. 2001), and long-term exposure may cause neurotoxicity and carcinogenicity (National Toxicology Program 2011). The plant alone is safe to use; however, it has been estimated that 2 to 20 cups of sage tea would be necessary to achieve the permissible daily consumption of thujone. Furthermore, thujone has a modest affinity for cannabinoid receptors but does not cause cannabinoid receptor agonism. 5-HT3 (ligand-gated ion channel serotonin) receptor activity has also been demonstrated to be reduced by thujone (Craft et al. 2017).

1.2.3 Basil

Basil (*Ocimum basilicum* L.) is an annual plant of the Lamiaceae family, known worldwide as a culinary and healthy herb (Lee & Scagel 2009). Basil's essential oils have been used in many fields for medicinal treatments, perfumery, and cooking spices. Basil is used extensively to add a distinctive aroma and flavour to food, such as salads, pizzas, meats and soups. Numerous studies have documented that basil contains high concentrations of phenolic compounds (especially rosmarinic acid and caffeic acid), which are characterised by high antioxidant capacity (Lee & Scagel, 2009; Surveswaran et al. 2007).

Many studies about the volatile composition of basil have been carried out. Basil essential oil contains mainly monoterpenes, sesquiterpenes, alcohols, aldehydes, ketones, esters and miscellaneous compounds (Lee et al. 2005). The most important compounds are affected by the geographical source, for instance linalool and estragole were dominant in Egyptian sweet basil (Karawya et al. 1974), linalool, estragole and eugenol in Israeli sweet basil (Fleisher 1981), eugenol, methyleugenol, eucalyptol and linalool in Italian sweet basil (Di Cesare et al. 2003) and Linalool and Eugenol in Spanish sweet basil (Díaz-Maroto et al. 2004).

1.2.4 Rosemary

Rosemary (*Rosmarinus officinalis* L.) of the family Labiatae, is an aromatic shrub with an intense pleasant smell reminiscent of pine wood. Rosemary is cultivated mainly in Mediterranean countries, such as Spain, Morocco, Tunisia, France and Italy (Flamini et al. 2002). The essential oil volatile composition of rosemary has been the subject of considerable research in recent years. Rosemary essential oil contains mainly monoterpenes and monoterpene derivatives (95–98 %), the remainder (2–5 %) being sesquiterpenes (Díaz-Maroto et al. 2007). The principal volatile compounds in rosemary are camphor and 1,8-cineole, followed by borneol, verbenone, α-pinene and camphene (Díaz-Maroto et al. 2007).

1.2.5 Ginger

Ginger (*Zingiber officinale* Roscoe) is one of the most well-known herbal medicines in Indonesia. This herbal is commonly used as raw material for traditional beverages, food seasoning, aroma therapy, and traditional medicines since years ago (Al Makmun & Widodo 2014). Ginger essential oil releases the specific odour and flavour that is distinguishable by human olfaction. The specific odour and flavour of ginger are generated by the combination of several major volatile compounds in ginger essential oil produced throughout the plant, especially at the rhizome. Different composition of major compounds generates different odour in ginger. Commonly, ginger odour exhibits a warm, sweet, bitter, and spicy taste with the slightly pungent mouth feel (Hardoyono & Windhani, 2019).

The rhizomes of ginger are characterised by a pungent flavour, resulting from the presence of gingerol compounds (Kumara et al. 2017), the most abundant of which are [6]-gingerol, [8]-gingerol and [10]-gingerol (Yudthavorasit et al. 2014). Furthermore, numerous

derivatives of gingerols are also present in fresh and dried ginger, including shogaols and paradols (Yudthavorasit et al. 2014). In combination with some of these derivatives, gingerols are reported to provide most of the documented medicinal properties of ginger (Kubra & Rao 2012), as well as its characteristic pungent taste and odour (Fisher & Scott 2007).

1.2.6 Thyme

Thyme is an aromatic medicinal plant of increasing economic importance for Europe, North Africa, and North America (Badi et al. 2004). The leaves can be used fresh or dried as a spice. Moreover, they can be used as herbal teas and condiments. EO extracted from fresh leaves and flowers can be used as an aroma additive in food, pharmaceuticals, and cosmetics (Hancı et al. 2003; Lee et al. 2005) and has been described as a storage alternative to chemical compounds due to its fungicidal ability in fruits (Valverde et al. 2005; Valero et al. 2006) Thyme also has different beneficial effects; for example, antiseptic, carminative, antimicrobial, and antioxidative properties (Shati & Elsaid 2009). The analytical studies performed on thyme show the main volatiles are Thymol, Carvacrol, p-Cymene, β-Pinene, γ-Terpinene, β-Caryophyllene, 1-Borneol, 1,8-Cineole (Hudaib & Aburjai 2007; Safaei-Ghomi et al. 2009).

1.3 Importance of Spices

Spices are used for several reasons, but the flavour is likely to be at the top of the list. Several spices just smell amazing, and their fragrances may be either calming or exciting. Ginger, on the other hand, is considered a stimulant, and the fragrance of ginger may be just what you need to lift your spirits. Certain spices have been thought to heal every ailment, disease, and affliction known to man throughout the years, but there is little doubt that spices have value that goes well beyond improving the flavour of food (Belay & Abisa 2015).

Spices have an important role in the nutrition of our regular diet. Scientists have undertaken significant research on this issue and determined that spices contain more antioxidants than fruits and vegetables. When spices are dried, they contain more antioxidants than when they are raw or fresh. Half a teaspoon of spices has more antioxidants than half a cup of fruits. Spices have an active role in the body by functioning as drugs. Cloves, oregano, allspice,

cinnamon, sage, peppermint, thyme, and lemon balm are among the spices used. These spices might provide a large nutritional source (Embuscado 2015).

Spices, the principal flavouring, colouring, and aromatic elements in foods and drinks, are becoming more popular as a result of their diverse uses. Spices' anti-diabetic, anti-hypercholesterolemic, anti-carcinogenic, and anti-inflammatory characteristics are crucial in today's world, since diabetes, cardiovascular disease, arthritis, and cancer are significant health issues. Spices or their active compounds may be used as possible treatments or preventatives for specific medical diseases (De & De 2019).

Extensive animal model research indicates that spices may be consumed at higher dietary levels without compromising growth, organ weights, food efficiency ratios, or blood components. Curcumin, the turmeric colouring pigment, capsaicin, the pungent principle found in red pepper, allicin, the active principle found in garlic, gingerol, the pungent principle found in ginger, saponin, and fenugreek fibre are all extremely valuable in health care due to their numerous physiological functions (Singh 2020).

Desiccation and freezing of food were not practical alternatives for people living in hot, humid areas until recently; these cultures discovered chemical preservation in the form of salt and spices. Spices were usually the sole alternate option for safeguarding food from insect infestation and microbiological putrefaction since the former was confined to certain places (Joardder & Masud 2019). Many of the highly aromatic phytochemicals that protect plants from insect and microbial assault are now discovered to be the same ones that "preserve" our bodies by protecting us from degenerative disease (Alamgir 2017).

Spices are used in significantly greater numbers and types in hot, humid climates than in colder climates. Spice consumption is greatest in India and Thailand, with the warm Mediterranean countries trailing behind these and other Eastern countries but ahead of the United States. Scandinavian nations consume the least amount of spices of any area. Furthermore, the importance of spices in the prevention of chronic degenerative sickness may be shown to correlate to the varying degrees of spice consumption that occur throughout temperature zones. Cold countries, which are often the most industrialised, have far higher incidence of chronic degenerative diseases than warm locations. Spices should be consumed

on a regular basis since they may help people feel better, think better, age slower, and fight diseases including cardiovascular disease, cancer, diabetes, Alzheimer's disease, and other chronic degenerative diseases (Shils 1999).

1.4 Volatile Compounds

Essential oil is the most volatile component of spices, even though it is present in minute amounts (Rao et al. 1998). According to the International Organization for Standardization (ISO), essential oil is a "product generated from a natural raw material of plant origin, by steam distillation, mechanical operations, or dry distillation, following separation of the aqueous phase — if any — by physical processes" (ISO 9235:2013). Essential oils have a broad variety of applications, including pharmaceutics, cosmetics, medicinal, and culinary applications (Orphanides et al. 2016).

Essential oils are maintained on the surface of fresh spice leaves in specialised structures known as trichomes, which are uni- or multicellular appendages in epidermal cells that extend outwards from the surface of plant organs such as leaves, roots, or barks (Werker 2000). The integrity of the dried product's oil glands is critical to the retention of essential oils in dried leaves (Ebadi et al. 2015). As a consequence, preserving trichome integrity or minimising trichome degradation during drying may boost essential oil output and aroma quality of dried spices. Because volatile compounds are water soluble and may accumulate in plant tissues, they can also be found in spices in glycosidic forms (Winterhalter & Skouroumounis 1997).

1.5 Spices drying methods

The drying technique is one of the most important variables influencing the quality of dried spices (Diaz-Maroto et al. 2002), and its impact has been widely researched. Because aroma compounds are heat-sensitive molecules that quickly evaporate from plant tissues during drying, high-temperature drying techniques would drastically reduce the amount of aroma compounds (Khangholil & Rezaeinodehi 2008). In contrast, the essential oil content of certain spices, such as Mexican oregano (shade, sun, and 40 $^{\circ}$°C were compared) (Calvo-Irabién et al. 2009) and bay leaf (convective drying at 40, 50, and 60 °C, sun drying, and

shade drying were compared), has been reported to be unaffected by the drying method studied (Demir et al. 2004).

Sun drying, shadow drying, freeze-drying, and hot-air-drying are all well-known spice-drying processes. Among these drying processes, hot-air oven-drying at temperatures ranging from 40 °C to 60 °C is the most commonly employed in spice drying research on a laboratory scale (Shaw et al. 2016). Because of the negative impact of high drying temperatures on the quality of dried products, numerous research has concentrated on the development of alternative drying processes that may offer advantages over conventional methods. Solar-assisted drying, microwave drying, microwave-vacuum drying, infrared-assisted drying, heat-pump drying, and contact drying have been used in industry (Thamkaew et al. 2021).

The impact of traditional and newly developed drying processes on the quality of dried spices is discussed below.

1.5.1 Sun drying

Sun or sunlight drying was the first drying procedure employed in most tropical or subtropical climates, and it is still used to dry many kinds of agricultural goods, such as medicinal plants and aromatic spices (Orphanides et al. 2016). During the drying process, fresh spices are placed on well-ventilated drying racks and exposed to direct sunlight (Janjai & Bala 2012). Sun drying may not be a practical drying process for certain spices due to low product quality. Sun drying drastically reduces the colour and aroma of dried spices.

In the case of roman chamomile, the amount of important volatile components such as isobutyl isobutyrate, 3-methylbutyl isobutyrate, and propyl tiglate was lower in sun-dried samples than in hot-air-dried samples (dried at 40 °C) (Omidbaigi et al. 2004). In the case of lemon grass, sun-dried lemon grass was shown to contain less total essential oil than hot-air-dried lemon grass (Hanaa et al. 2012). Sun drying at 40 °C resulted in a greater loss of essential oil content in basil (*Ocimum basilicum* L.) than shade or hot-air-drying (Hassanpouraghdam et al. 2010). Sun drying caused higher damage to the epidermal surface, shortening of the glandular trichomes, and a bigger loss in mineral content in *Vernonia amygdalina* leaves than shade drying (Alara et al. 2018).

1.5.2 Shade drying

Shade drying is another spice drying method that employs the sun's energy as a heating source. The method is similar to sun drying in that the spices are put in the shade in a room with good ventilation, low humidity, e.g., 22–27 % for *Lippia citriodora* (Ebadi et al. 2015), and no direct sunlight exposure. During the shade-drying process, the ventilated air is heated using sun energy before passing through the spices (Sharma et al. 2009). This drying method may be superior to sun drying due to its ability to preserve light-sensitive chemicals and minimise light-induced chemical reactions such as oxidation. Shade drying, on the other hand, takes longer than sun drying, which is already considered an extremely prolonged technique (Pirbalouti et al. 2013).

For several kinds of spices, including rosemary, shade drying has been demonstrated in research to be a superior drying technique in terms of retaining essential oil content and colour of dried goods when compared to other drying methods such as hot-air-drying, sun drying, microwave drying, and freeze-drying (Khorshidi et al. 2009). Shade drying of *Tanacetum parthenium* (compared to oven-drying at 40 °C and sun drying) (Omidbaigi et al. 2007), thyme (compared to freeze-drying) (Sarosi et al. 2013), basil (compared to oven-drying at 40 and 60 °C and sun drying) (Hassanpouraghdam et al. 2010), mint (compared to convective drying at 40 °C) (Rababah et al. 2015), lemon balm (compared to convective drying at 40 °C) (Rababah et al. 2015), and sage (compared to convective drying at 40 °C) (Rababah et al. 2015).

1.5.3 Solar-assisted drying

Solar-assisted drying is a modification of the well-known process of sun drying. Researchers have concentrated their efforts on creating unique sun-assisted drying technologies due to the fact that solar energy is free. This improvement intends to increase the drying process' energy efficiency while addressing the major shortcomings of conventional sun drying. Direct sunlight drying (also referred to as sun drying in this research), indirect solar drying (also known as convective solar drying), and mixed-mode or hybrid solar drying are the three methods of solar drying (Rabha et al. 2017). Several studies on the development of solar-assisted spice dryers have been conducted in recent years, including forced convection solar

tunnel dryers (Rabha et al. 2017), forced convection solar greenhouse dryers (Morad et al. 2017), solar-assisted fluidized bed dryers (Ceylan & Gurel 2016), and solar collector dryers (Sevik 2014).

1.5.4 Hot-air-drying

As previously noted, the fundamental downside of solar-powered drying procedures is that they take an unusually long time to dry. Oven-drying (also known as "convective drying" or "hot-air-drying") is the most common and widely used spice drying method, particularly in non-tropical climates where sun and shade drying are inadequate (Orphanides et a. 2016). One of the main advantages of hot-air-drying is its controllability, which allows food manufacturers to have total control over process parameters, including drying temperature, drying duration, and air velocity. These parameters may be modified to acquire the desired product qualities (Orphanides et al. 2016). Process parameters for different spices have been examined and altered in order to increase dried product quality (Orphanides et al. 2016).

The total volatile chemical concentration is low after hot air-drying, however (Chua et al. 2019). Hot-air-drying may impair spice scents, and high drying temperatures can lead to pigment loss (Fennell et al. 2004). As a consequence, moderate drying temperatures (35–50 °C) have been suggested for the preservation of heat-sensitive components in dried items (Muller et al. 1989). The increased evaporation of moisture and volatile compounds due to hot air flow over the materials during the drying process creates an ideal environment for oxidation activities (Orphanides et al. 2016). Product shrinkage and high energy use are other important drawbacks of hot-air-drying (Orphanides et al. 2016). Furthermore, since hot-air-drying is one of the most energy-intensive food processing methods, efforts have been focused on reducing energy consumption, increasing process efficiency, and reducing drying time (Won et al. 2015).

1.5.5 Freeze-drying

Numerous studies have suggested freeze-drying as a useful drying procedure for keeping the fresh-like aroma of spices due to its low operating temperature (Antal 2010). In several kinds of spices, including spearmint, this drying approach has been thoroughly documented to produce dried spices with better fragrance compared to other drying procedures, with lower

aroma component loss compared to hot-air-dried leaves (Antal et al. 2011). When basil leaves were freeze-dried vs air-dried at 50 °C, equal results were found (Di Cesare et al. 2003). Sun drying, shade drying, hot air-drying at 40 and 60 °C, and microwave drying at 500 and 700 W all failed to retain the yield and chemical composition of the essential oil of purple and green basil leaves (Pirbalouti et al. 2013). Coriander from Iran had similar results (Pirbalouti et al. 2017). The concentration of total volatiles in freeze-dried thyme was only decreased by 1–3 %. (Venskutonis et al. 1996).

Freeze-drying, on the other hand, yields lower-quality dried items as compared to microwave drying. Freeze-dried leaves of garden thyme (*Thymus daenensis*) had large amounts of essential oils and were of acceptable colour but had a weaker aroma than microwave-dried leaves (Rahimmalek & Goli 2013). Freeze-drying delivers high-quality dried spices in terms of bioactive components in various kinds of spices. When thyme leaves were freeze-dried and hot-air-dried, freeze-dried thyme leaves yielded more thymol than oven-drying at 30-500 °C and shade drying (Sárosi et al. 2013).

1.5.6 Microwave drying

Microwave drying is a drying technology that is increasingly being employed in the spice industry (Moses et al. 2014; Wray & Ramaswamy 2015). It allows for rapid evaporation of water from food, resulting in shorter drying times as compared to other drying methods (convective drying, shadow and sun drying, and freeze-drying) (Chi et al. 2003) and reduced energy use throughout the drying process (Di Cesare et al. 2003). Microwave-dried items showed less shrinkage, better colour, and rehydration capability as compared to hot-air-drying (Kathirvel et al. 2006). Microwave power (W), drying time, and beginning moisture level are important drying variables that influence the quality of microwave-dried items (Moses et al. 2014).

Microwave drying creates high-quality dried items by retaining or enhancing the quantity of bioactive components. Microwave drying at 850 W resulted in better trans-b-carotene intactness and pigment extractability in dried coriander leaves than convective drying at 450 °C. (Divya et al. 2012). Similar findings were found with microwave-dried sage leaves at 850 W (Hamrouni-Sellami et al. 2013), where the microwave dried product preserved greater

total phenolic components, flavonoid content, and antioxidant activity than the dried items produced by convective drying at 45 °C. The leaves of *Gynura pseudochina* yielded similar findings (Sukadeetad et al. 2018).

Microwaves may be used in combination with other drying processes such as hot air-drying, either as a pre-drying step to reduce the initial moisture content of the materials or as the last stage of drying (Orphanides et al. 2016). The primary drawback of microwave drying is the non-uniformity in heating, which results in temperature variations in the product during the drying process, particularly in large-size items. This uneven heating may result in uneven drying of the goods, overheating, and quality degradation (Ozkan et al. 2007).

Nonetheless, there has been a surge in interest in microwave drying spices in recent years. This is most likely because spices are frequently smaller and thinner in size than most other solid foods, and hence non-uniform heating may not be a major drawback for microwave drying of spices. Microwave drying, on the other hand, has been demonstrated to result in a larger reduction of smell components in certain herbs, such as marjoram (Raghavan et al. 1997) and rosemary (Rao et al. 1998) as compared to other drying techniques, such as convective drying, shadow drying, and sun drying. When compared to other traditional drying procedures, microwave drying time is much quicker. More study is required, however, for additional spices in order to improve the quality of the dried items and optimise the process (Moses et al. 2014).

1.5.7 Microwave-vacuum drying

Microwaves in combination with vacuum drying have recently gained popularity (Orphanides et al. 2016). Microwave irradiation is utilised as a heating source in the sub-atmospheric pressure drying chamber to boost the temperature of the food products. In contrast to convective and microwave drying, the vacuum provides the driving force for water evaporation, resulting in faster drying rates (Soysal 2004). Microwave-vacuum drying, as opposed to hot-air-drying, may reduce drying time by 70–90 % while creating higher-quality items (Giri & Prasad 2007). Thymol levels in vacuum-microwave dried *L. Berlandieri* were 1.3 times higher than in air-dried *L. Berlandieri* (Yousif et al. 2000).

Microwave-vacuum drying of mint leaves resulted in greater colour retention of dried items as compared to hot-air-drying. SEM pictures of microwave-vacuum dried items exhibited increased porosity and less collapse when compared to hot-air-dried samples (Therdthai & Zhou 2009). The vacuum pump's capacity, on the other hand, is the major restriction of the vacuum drying process. Because of the high initial moisture load from food, the vacuum pump's capacity may be quickly surpassed, resulting in less efficient functioning.

1.5.8 Heat-pump-assisted drying

Heat-pump drying is another drying technology advancement that aims to increase the efficiency of traditional convective drying. In most cases, a heat pump is utilised in conjunction with another airdrying equipment to boost the temperature of the original input air. The technology is also known as a "heat pump dryer" or a "heat pump-assisted drier" (Fatouh et al. 2006). The heat pump drier is perfect for industrial spice drying since it can operate at a broad range of air velocity and drying temperatures (Fatouh et al. 2006). Another key benefit of a heat pump drier is its ability to dehumidify the exit air of the drying unit. When the evaporator temperature is lower than the dew point of the air near the evaporator intake, the dehumidifying action occurs (Fatouh et al. 2006).

Heat-pump drying may deliver higher-quality dried items due to its ability to adjust the properties of the air throughout the drying process. When compared to standard solar greenhouse dryers, heat pump sun drying of java tea (*Orthosiphon aristatus*) displayed enhanced controllability of the drying room's relative humidity, particularly at night. The dehumidifying system reduced the drying chamber's relative humidity by 10–15 % while maintaining the maximum relative humidity of 65 %. Furthermore, the drying rate of the heat pump-integrated solar greenhouse was 3-4 times quicker than that of a standard greenhouse drier (Tham et al. 2017).

1.5.9 Infrared drying

This drying method's versatility, simplicity, rapid heating rate, and quick drying rate are all significant advantages (Ashtiani et al. 2017). During the process, electromagnetic energy from infrared wavelength radiation is transferred and absorbed by the material, resulting in heat generation from within the material due to changes in the molecule vibrational state

(Krishnamurthy et al. 2008). Infrared drying is more energy efficient than hot air-drying. However, just a few infrared spice drying studies have been conducted in recent years. When drying mint leaves (drying temperatures of 30, 40, and 50 °C were examined), infrared drying exhibited a higher energy efficiency and drying rate than convective drying (Ashtiani et al. 2017).

When the infrared drying temperature was enhanced, the quantity of crocin and safranal in dried saffron increased (Torki-Harchegani et al. 2017). These are the key chemical components that contribute to the quality of dried saffron. Infrared irradiation is appropriate for thin-layer drying due to its short travel distance through the materials and the dependency of the contacted area on the materials. Furthermore, the quick drying rate (when compared to hot-air-drying) (Ashtiani et al. 2017; Torki-Harchegani et al. 2017) and capacity to sustain a high drying rate at lower moisture content (Paakkonen et al. 1999) of infrared drying make it a viable alternative drying technique for spices. However, Chua et al. (2019) discovered that non-uniform infrared heating reduces the scent quality of dried spices.

1.5.10 Fluidized bed drying

In the food industry, many different agricultural items, including spices, have been dried in fluidized beds (Gangopadhyay & Chaudhuri 1979). The method is carried out by directing high-velocity hot air to the drying bed upon which the objects are placed (high enough to cause fluidization of the products). The drying rate of this technology is much higher than ordinary convective drying because of the increased heating rate of the fluidization heating. When drying lemon myrtle leaves in a fluidized bed, increasing the drying temperature (drying temperatures of 30, 40, and 50 °C were studied) resulted in increased citral content retention (which contributes to the "citrus" scent) of the dried product (Buchaillot et al. 2009). The lowest observed drying temperature (30 °C) resulted in higher colour retention, while the maximum measured drying temperature (50 °C) resulted in unacceptable colour quality loss.

Spices may not be appropriate for fluidization drying due to their high moisture content, large surface area to volume ratio, and rough surfaces, which may result in poor air percolation. Vibrofluidized bed drying was created to overcome this problem (de Aquino et al. 2017).

The vibrofluidized drying technique is a sort of fluidized bed dryer that contains a vibrator module to improve the performance of the fluidized bed dryer. Unlike typical fluidized bed drying, vibrofluidized bed drying met the moisture reduction and homogeneity criteria for dried basil leaves. However, at drying temperatures of 45 and 60 °C, the dried product's eugenol content declined.

1.5.11 Supercritical CO_2 drying ($scCO_2$)

In this method, supercritical carbon dioxide is employed as a drying medium. The key benefits of this drying method are its low working temperature (usually around ambient), absence of oxygen, limited product shrinkage, and better rehydration capacity of dried products. There have only been a few studies on the drying of spices using $scCO_2$. CO_2 drying of basil was observed to be superior to other drying processes such as convective drying (40 °C for 26 hours) and freeze-drying (Busic et al. 2014). When colour, bioactive components, and fresh-like features were investigated, the findings showed that freeze-drying gave the highest dried basil quality, followed by $scCO_2$ drying, and convection drying generated the lowest dried product quality.

However, it was determined that $scCO_2$ drying was the best drying approach among the three investigated drying procedures due to the acceptable quality of the dried spices and considerably quicker drying time (2–3 h) compared to freeze-drying (4 days) and air-drying (26 h). Another study was conducted on $scCO_2$ drying of spices in combination with ultrasonic pre-treatment of coriander leaves (Michelino et al. 2018). Bacteria were successfully inactivated by $scCO_2$ drying, according to the findings. According to the data, yeast, moulds, and mesophilic bacteria were reduced by 4 logs throughout the drying process.

1.5.12 Radio-frequency drying

Radio-frequency drying (RFD) combines radio frequency heating with convection drying. Radio frequency heating, like microwave heating, is based on the dielectric properties of food components but at distinct wave frequencies (Nijhuis et al. 1998). Radio-frequency heating might help increase the drying rate, especially during the declining rate phase, when typical convective drying is restricted (Thomas 1996).

The impact of infrared RF drying on dill greens quality was compared to convective drying (Naidu et al. 2016). RF drying surpassed convective drying at 50 °C. However, as compared to convective drying (50 °C, with 58–63 % RH and 28–30 % RH) and infrared drying, RF-dried dill greens had the lowest bioactive component content (containing chlorophylls a and b, carotenoids, and ascorbic acid). Given the loss of chlorophyll and the resulting colour changes, the findings imply that RF drying may not be a suitable spice drying approach.

1.5.13 Hybrid drying methods

Hybrid drying techniques overcome the issue of single stage drying by integrating two or more drying processes. We investigated heat pump drying, solar assisted drying, microwave-vacuum drying, and radiofrequency drying in this study. Because of their potential to shorten processing time, avoid quality degradation, and maintain process efficiency, these drying techniques have recently piqued the attention of academics (Chou & Chua, 2001). Solar-assisted drying, microwave-assisted drying, and heat pump-assisted drying are currently the three technologies that have received the most attention (Chou & Chua 2001; Jin et al. 2018). However, nothing is known about the influence of these hybrid processes on the quality of dried spices.

1.6 Changes in Volatile compounds during Drying

Spice volatile compounds are composed of a few or many chemical components, with some spices including over a hundred chemical compounds (Antal et al. 2011). Essential oils' chemical composition varies depending on the spice, harvesting season, postharvest techniques, plant age, and storage conditions (Dokhani et al. 2005). Each chemical component gives the essential oil a particular flavour. This contribution is based on their distinct odour threshold, which is determined by the structure and volatility of the chemical (Turek & Stintzing 2013). Even with minor components, changes in the quantity of essential oil chemical components (either via chemical reactions or degradation) may cause major differences in essential oil flavour (Grosch 2001).

The chemical components of essential oils are unstable. Chemical reactions such as oxidation, isomerization, cyclization, and dehydrogenation may readily convert them into different types of molecules. These chemical reactions may begin chemically or

enzymatically (Turek & Stintzing 2013). One of the most significant chemical changes in essential oil components is autoxidation. The autoxidation process affects the decomposition of terpenoids, the most abundant natural volatile class in plants (Baser & Demirci 2011). Secondary products, such as hydroperoxides, may be formed during the autoxidation of terpenoids and then destroyed in the presence of light, heat, and acid during later stages of the oxidation process (Turek & Stintzing 2013).

These chemical changes in the essential oil components might occur during the drying process or when the dried items are stored. The use of heat while drying might hasten these chemical processes (Lee, Lee & Choe 2007). During the drying process, heat induces the initial generation of free radicals, which catalyses the essential oil autoxidation mechanism (Choe & Min 2006). As a result of the higher drying temperature, there is a greater loss of aroma compounds and, as a result, a deterioration in the quality of the loss of aroma in dried spices.

The fall or modification of volatile components in dried spices relies on drying parameters such as drying method, temperature, vacuum (for example, for drying or freezing), drying duration, and the amount of water lost during drying (Antal 2010; Figiel & Michalska 2016). In general, drying spices decreases volatile compounds, and certain drying procedures maintain volatile components better than others (Chua et al. 2019).

The drying temperature is critical in preserving the volatile components of dried spices. The drying process's high temperature frequently results in the loss of volatile compounds. Trichomes may be dried at high temperatures, resulting in the loss of volatile chemicals due to evaporation. Furthermore, the degradation of heat safety compounds in essential oils may result in high drying temperatures (Argyropoulos & Muller 2014).

Another important factor in essential oil manufacturing is the vacuum level (Chua et al. 2019). Although the pressure in the chamber reduced in the case of freeze-dried spearmint, it also resulted in a significant loss of volatile compounds (Antal et al. 2011). The quality of volatile components of dried rose tubes decreased with increasing vacuum levels during vacuum-microwave drying (Calín-Sánchez et al. 2011).

The amount of moisture lost from the tissue is another factor influencing the volatile components of dried spices. The quantity of water evaporated in air-dried oregano was directly related to the reduction in volatile chemicals because water vapour may act as a transporter for the diffusion of volatile tissue components into the environment during the drying process (Figiel et al. 2010). Furthermore, volatile compounds with a high-water affinity are more likely to be lost during drying (Sellami et al. 2011).

Changes in volatile compounds in spices throughout the drying process are also affected by biological factors such as beginning humidity content, plant age, growth conditions, and harvest timing (Ascrizzi et al. 2018). The volatile concentration of dried items is also affected by storage conditions, particularly when light and oxygen are present (Baritaux et al. 1992). Reducing essential oil components such as pulegone and hepatotoxin may be advantageous, as found in *Hedeoma pulegioides* and *Mentha pulegium* (Asekun et al. 2007). Hot air-drying at 40 °C significantly reduced the pulegone content of dried mint (*Mentha longifolia* L. subsp. capensis) (Chen et al. 2001). As a result, it is preferable to consume this kind of mint dried rather than fresh.

1.7 Extraction Methods

1.7.1 Conventional extraction methods

The bulk of traditional extraction processes depend on the ability to extract different solvents as well as the ease with which heat and/or mixing may be applied. The most common procedures are Soxhlet extraction, maceration, hydro distillation, and steam distillation.

1.7.1.1 Soxhlet Extraction

This process for extracting lipids from solid materials was invented by German scientist Franz Ritter von Soxhlet in 1879. It is currently often used as a standard approach for measuring the lipid extraction yield from plant sources. Soxhlet extraction works by dissolving oil-based compounds within a thimble-sized plant sample using a solvent vapour. The vapour is subsequently condensed, as shown in Figure 1, to separate the compounds of interest from the solvent. Although Soxhlet extraction is simple, inexpensive, and easy to use, it requires a large amount of solvent, a lengthy extraction duration, and causes heat-labile bioactive compounds to deteriorate (Ngamwonglumlert et al. 2017).

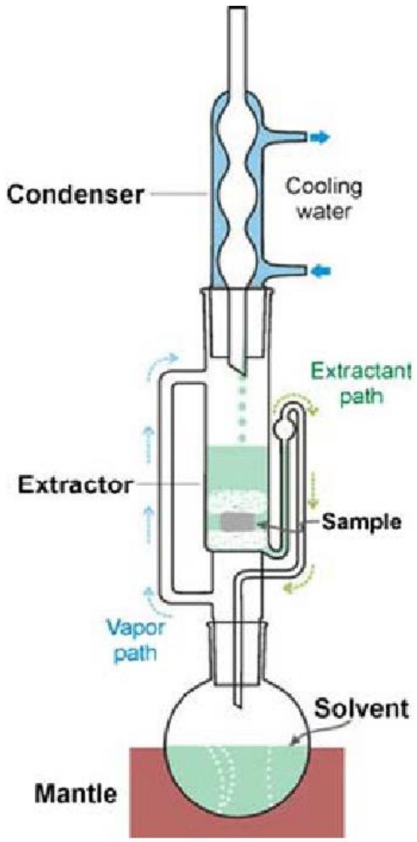

Figure 1: Soxhlet extraction setup (Source: Rassem et al. 2016)

1.7.1.2 Maceration

Maceration is a simple extraction method that does not need any additional heat. To increase the exposure of bioactive components to the solvent, a plant sample is generally crushed and mixed with the solvent of choice. The mixture is left in an extraction jar, sometimes stirred. After the treatment is completed, the liquid is separated from the solid using a mechanical press or centrifuge. Because this procedure is often used at room temperature, a long extraction time is required. Furthermore, a substantial amount of solvent is necessary to repeat the extraction until no more interesting compounds are present in the sample (Azmir et al. 2013). One of the main drawbacks of this method is that the extract is acquired in its raw form, requiring subsequent purification.

1.7.1.3 Hydrodistillation

Hydrodistillation has been used for many years to extract essential oils and bioactive compounds from plant sources. The three ways of hydrodistillation are water distillation, water and steam distillation, and straight steam distillation (Azmir et al. 2013). The three basic physicochemical processes involved in hydrodistillation are hydro-diffusion, hydrolysis, and heat decomposition. The principal channels for releasing and transferring bioactive compounds from the plant matrix are hot water and steam. The vapour mixture is condensed by indirect cooling, enabling oil and bioactive compounds to separate from the water. In the setup, a condenser and a decanter are also employed to collect condensate and extract essential oils from water, respectively (Figure 2). Essential oils and oil-based bioactive compounds are typically dried over anhydrous sodium sulphate. Certain volatile components, natural hues, and heat-labile bioactive compounds may be lost during hydrodistillation since it is typically conducted at temperatures greater than the boiling point of water.

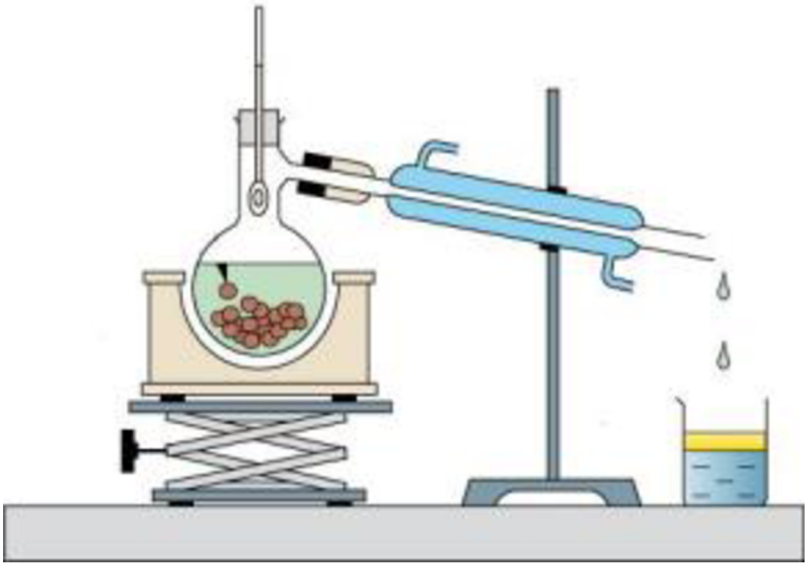

Figure 2: Schematic of hydro distillation technique (Source: Azmir et al. 2013)

1.7.1.4 Steam Distillation

Steam distillation is a kind of distillation (a separation or extraction process) that is employed for temperature-sensitive plants such as natural aromatic compounds. It was formerly a

standard laboratory process for purifying organic compounds, but vacuum distillation has made it obsolete. Steam distillation is still employed in a variety of industrial applications (Fahlbusch et al. 2003). Steam distillation is one of the most ancient and extensively used methods for obtaining essential oils from plant sources. Without being macerated in water, the plant materials charged in the alembic are exposed to steam. From the bottom to the top of the alembic, the injected steam flows through the plants.

Steam distillation, as seen in Figure 3, is a process in which steam travels through the material. This steam serves as an agent, breaking up the pores of the raw material and allowing the essential oil to be released. The technology generates a vapour as well as the necessary essential oil. This vapour is then condensed further, and the essential oil is extracted (Rai & Suresh 2004). The essential theory behind this method is that when the combined vapour pressure reaches roughly 100 °C, it matches the ambient pressure, enabling volatile components with boiling points ranging from 150 to 300 °C to be evaporated at temperatures close to water. In addition, depending on the intricacy of the essential oil extraction, this technique might be conducted under pressure.

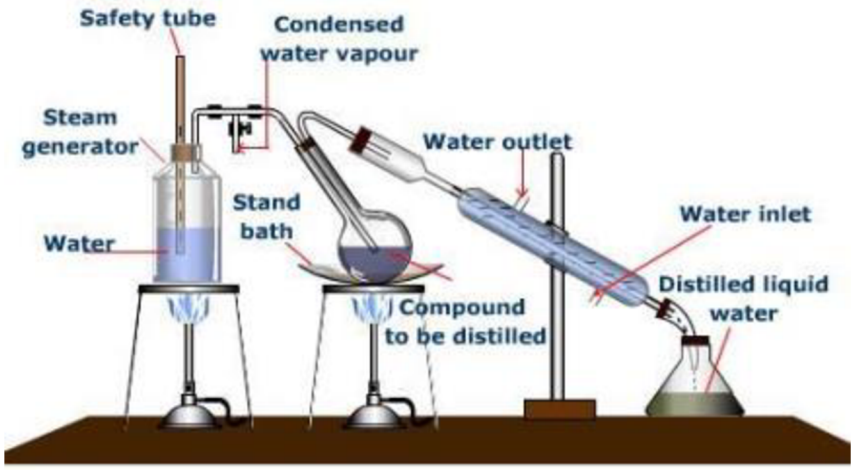

Figure 3: Schematic of steam distillation technique (Source: Fahlbusch et al. 2003)

1.7.2 Novel Extraction Methods

1.7.2.1 Enzyme-Assisted Extraction

The majority of bioactive chemicals found in plants are either disseminated throughout the cytoplasm, which is bordered by cell membranes and cell wells, or are imprisoned in the

polysaccharide-lignin network by hydrogen or hydrophobic bonding, rendering them inaccessible to the solvents employed in standard extraction methods. The addition of specific enzymes during extraction such as pectinase, cellulose, amylase, and hemicellulose facilitates the release of cellular constituents and improves extraction recovery by breaking down cell walls and membranes and hydrolyzing polysaccharides and lipid complexes, including membranes (Azmir et al. 2013).

Enzymes have been created using fungi, bacteria, animal organs, and fruit/vegetable extracts. They are usually used to prepare plant materials before standard extraction. The EAE technique has been developed as a novel and ecologically friendly way for releasing bound molecules and enhancing overall yield. EAE's advantages include its nonthermal nature, reduced solvent and energy consumption, lesser toxicity, and aqueous solution effectiveness (Puri et al. 2012).

To effectively use enzymes for extraction enhancement, it is necessary to understand their catalytic property and mode of action, optimal operating conditions, and which enzyme or enzyme combination is suited for certain plant material. The efficiency of EAE is determined by enzyme composition and concentration, plant particle size, solid-to-water ratio, and hydrolysis time (Niranjan & Hanmoungjai 2004).

1.7.2.2 Supercritical Fluid Extraction (SFE)

Supercritical Fluid Extraction (SFE) exposes a specific gas to pressure and temperature levels that surpass its critical point, where separate gas and liquid phases do not exist. Supercritical fluid has the density and solvation power of a liquid, as well as the diffusion, viscosity, and surface tension of a gas. Because of these properties, it is an excellent solvent for extracting flavour and bioactive components from plant materials in a short amount of time and with higher yields at low temperatures. As pressure decreases, their solvation power increases (Sowbhagya & Chitra 2010). SFE has various benefits over other extraction procedures, including the absence of solvent residues and the strong top flavour notes of the extracts. A single component may be removed selectively using the right process conditions. The key criteria affecting extraction efficiency are temperature, pressure, particle size and moisture

content of raw material, extraction time, gas flow rate, and solvent-to-raw material ratio (Azmir et al. 2013).

Carbon dioxide (CO_2) is an appropriate solvent for SFE since its critical temperature (31 °C) is close to room temperature and its critical pressure (74 bars) is low, enabling it to work at moderate pressures, as illustrated in Figure 4. The SFE is often utilised at temperatures ranging from 30 to 60 °C and pressures ranging from 8 to 40 MPa (Ngamwonglumlert et al. 2017). The only disadvantage of CO_2 is that it has a low polarity, which makes it unsuitable for the extraction of polar molecules, which are common in pharmaceuticals and drug samples. This constraint has been efficiently bypassed by the use of chemical modifiers such as dichloromethane (CH_2Cl_2). A small amount of modifier may generally significantly enhance the polarity of carbon dioxide (Hawthorne et al. 1994). Although SFE is very successful in extracting heat-labile bioactive compounds and requires little solvent, the high pressure required for the process mandates a high capital and operational expense.

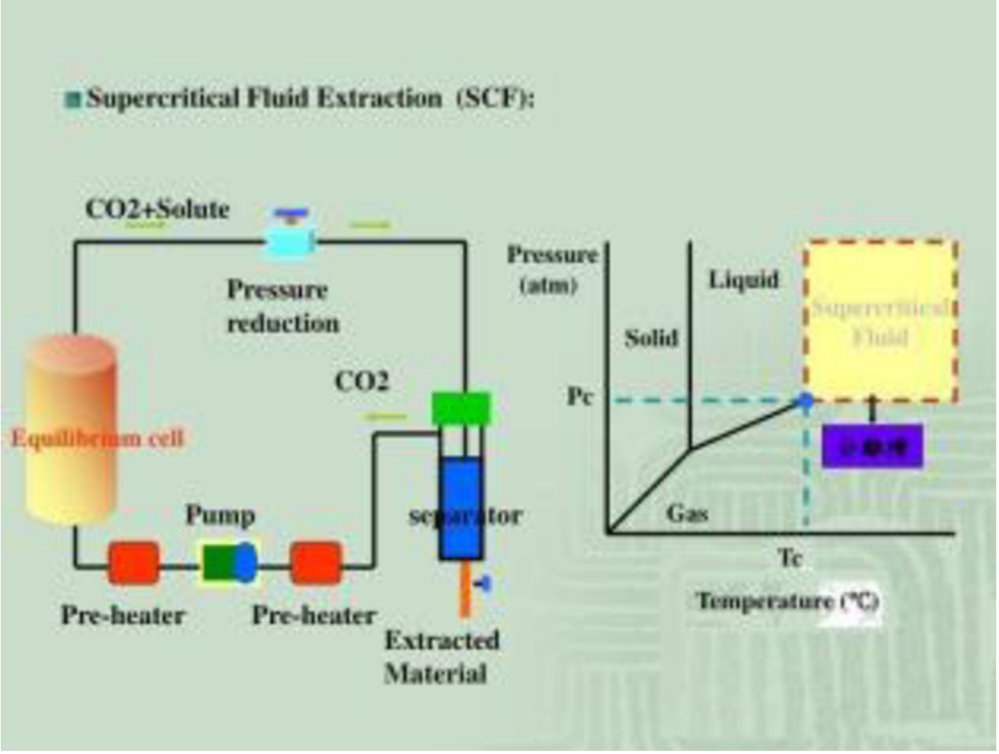

Figure 4: Schematic of Supercritical Fluid Extraction technique (Source: Ngamwonglumlert et al. 2017).

1.7.2.3 Pressurized Liquid Extraction

The aim of PLE is to employ high pressure to keep solvents liquid after they have boiled. Pressure and temperature ranges are typically 10.3–13.8 MPa and 40–200 °C, respectively. High pressure forces the solvent into the pores of the sample matrix, allowing for more interaction between the solvent and the compounds to be extracted. High temperature, on the other hand, causes more solvent penetration into the sample matrix while also disrupting plant cells and increasing solubility and mass transfer rate. As a consequence, PLE may significantly reduce extraction time and solvent use. Depending on the solvent, the PLE may extract both water- and oil-based compounds effectively. For polar chemical extraction, PLE is regarded to be a preferable option to SFE (Azmir et al. 2013). However, since the sample is subjected to high temperatures, PLE is ineffective for extracting heat-labile compounds. One of the key drawbacks of PLE, as well as SFE, is the high capital and operating costs involved with the use of high-pressure extraction.

1.7.2.4 Ultrasound-Assisted Extraction

The UAE goal is to use ultrasonic bubble cavitation (20 kHz to 100 MHz) to break plant cell walls and whole plant components, enhancing extraction efficiency. Cavitation is a phenomenon caused by an ultrasonic wave passing through a liquid medium, causing bubbles to alternate between compression and expansion cycles (Figure 5). When bubbles get too large to be confined by surface tension, they collapse, resulting in considerable shearing force and a massive amount of energy being transferred from kinetic energy of motion to heating of the bubble content. According to Suslick & Doktycz (1990), the bubbles have a temperature of over 5000 K, a pressure of roughly 1000 atm, and a heating and cooling rate of more than 1010 K/s. Cavitation, according to this theory, occurs only in liquids and liquid-containing solids. The UAE may effectively increase organic and inorganic components leaching from the plant matrix.

The moisture level of the sample, milling degree, particle size, and kind of solvent employed all have a significant impact on extraction efficiency. Furthermore, the essential process factors for the action of ultrasound are temperature, pressure, frequency, and time of sonication. The UAE has also been used with other approaches in order to improve the efficiency of a traditional system. The capacity to extract at lower temperatures owing to the absence of external heat input, minimal solvent consumption, and reduced extraction time are all advantages of UAE. Ultrasound energy can also help with quicker energy transfer, more effective mixing, smaller thermal gradients and extraction temperatures, selective extraction, and decreased equipment size.

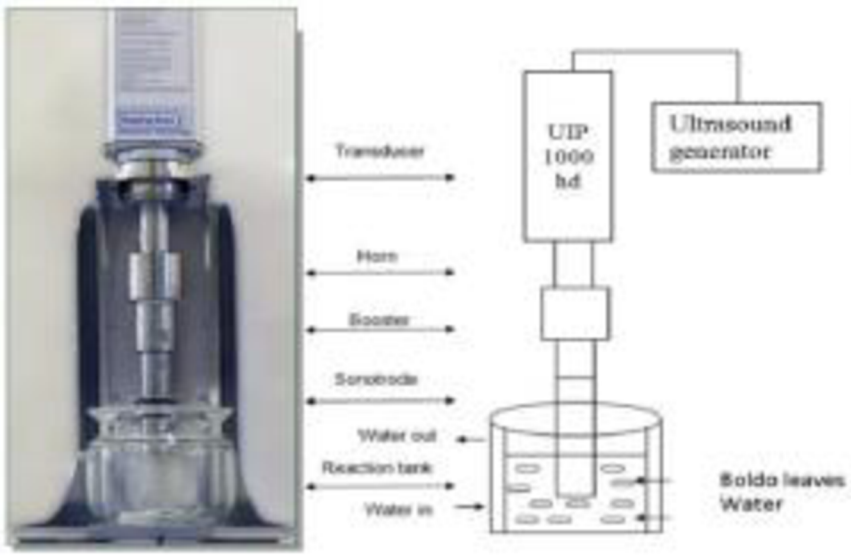

Figure 5: Schematic of Ultrasound-Assisted Extraction technique (Source: Suslick & Doktycz 1990)

1.7.2.5 Microwave-Assisted Extraction

The MAE is a novel technique that uses microwave radiation to extract soluble compounds from plant materials into fluids (both polar and nonpolar). Microwaves are electromagnetic fields with frequencies that range from 300 MHz to 300 GHz. As shown in Figure 6, fast heating is produced by ionic conduction and dipole rotation processes, resulting in rapid expansion of cell structure and rupture of plant cell walls and membranes. These mechanisms quicken bulk movement and extraction. MAE has a high extraction yield, a short processing time, a small temperature gradient, a low solvent consumption, and a tiny extraction unit (Azmir et al. 2013). However, it is not suitable for the extraction of heat-labile compounds. MAE in combination with vacuum has recently been presented as a method of functioning at lower temperatures (Ngamwonglumlert et al. 2017). MAE efficiency is affected by solvent type and concentration, extraction period, and microwave power.

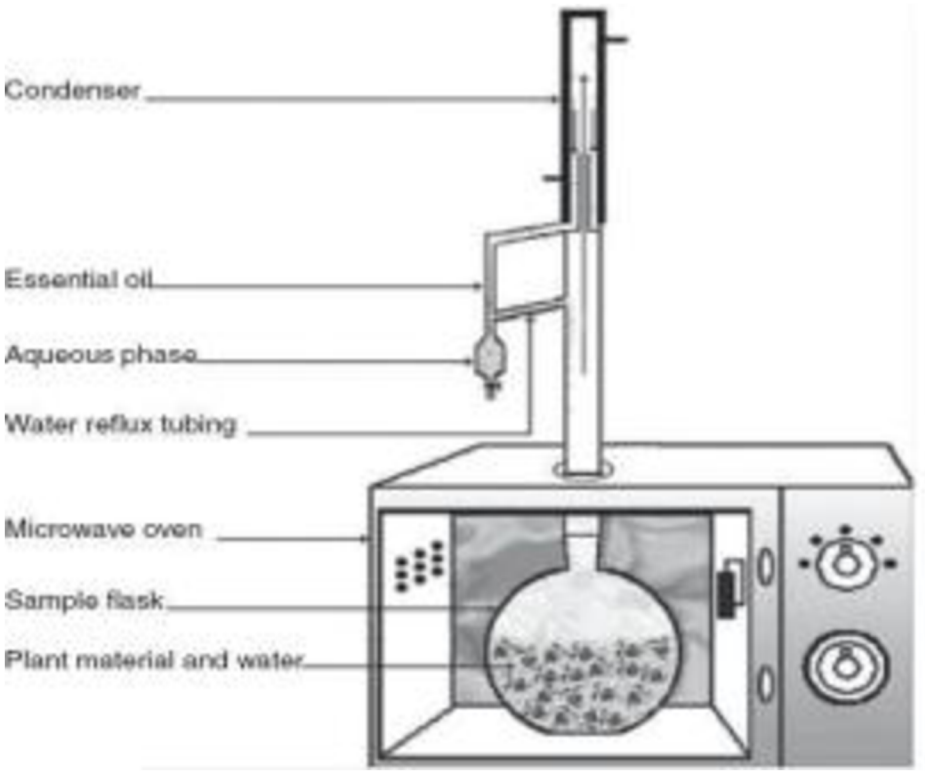

Figure 6: Schematic of Microwave-Assisted Extraction technique (Source: Azmir et al. 2013)

1.7.2.6 Pulsed Electric Field Extraction

Many mass transfer processes have benefitted from pulsed electric field (PEF) pre-treatment, including drying, diffusion, pressing, and extraction. When a short and high-voltage electric field is produced, the electroporation effect may induce nonthermal rupture of biological membranes (Asavasanti et al. 2011). Based on their charges, molecules segregate and accumulate on opposite sides of lipid bilayer membranes after PEF treatment. When the transmembrane voltage surpasses a threshold of roughly 1 V, holes form in the membrane's weak area, resulting in an increase in electrical conductivity and cell membrane permeability (Azmir et al. 2013). PEF may therefore be used to increase extraction yield while minimising extraction time.

PEF efficiency is affected by electric field strength, specific energy input, pulse number, frequency, treatment temperature, and sample properties. PEF has many advantages,

including a quick treatment time and little to no heat production, which allows for the extraction of heat-sensitive compounds. Nonpolar molecules, on the other hand, do not benefit from PEF treatment since this method necessitates the use of a solvent with high electrical conductivity (Ngamwonglumlert et al. 2017). Furthermore, PEF demands a high-power supply generator and a particularly built treatment chamber, making industrial-scale implementation expensive and difficult.

1.7.2.7 Gas chromatography/mass spectrometry (GC/MS)

GC/MS covers the identification/quantification of a large variety of volatile as well as non-volatile metabolites (following derivatization), mainly those involved in primary metabolism, including organic and amino acids, sugars, sugar alcohols, and phosphorylated intermediates (in the polar fraction of extracts) as well as lipophilic compounds such as fatty acids and sterols (in the a polar fraction) (De Vos et al. 2007). Most volatile compounds, being of low molecular weight, are often detected in the mass range of 30–400 m/z. However, when detecting specific high molecular weight metabolites, the mass scanning could be over a wide spectrum of 30–600 m/z. Considering that GC evolved as a popular technique almost six decades ago and given the standardization methods using the retention index and Kovats index, it does not come as a surprise that there are well-established databases for identification of the mass spectrum obtained from GC/MS. Fragmentation in GC/MS can be carried out by electron impact or chemical ionization in positive or negative mode. Typically, the ionization energy supplied in GC/MS studies is stronger compared to those for LC/MS, and this leads to a large number of fragment ions, which nevertheless are amenable to an easy match with compounds in the National Institute of Standards and Technology database (deconvolution approaches may be used). One important point to be noted is that for analysis of sugars in seaweeds by GC/MS, the sugars are often reduced and per acetylated. However, this method does not allow differentiating between mannose and mannitol because all sugars are reduced to their corresponding sugar alcohols and reacted with acetic anhydride to yield volatile sugar derivatives, which could subsequently be analysed using GC/MS (Van Hal et al. 2014).

The most important method for characterising components in EOs research is GC/MS. When acquiring mass spectra from schedule analysis of compounds with pertinent data, GC/MS proved most beneficial. There are compounds with identical chemical formulae but different types of structure, such as many types of monoterpenes. The mass spectra of a higher number of isomers may be quite similar. After mastering component elution time and integrating it with MS, the data quality increases correspondingly and becomes incredibly valuable. Combining IR spectroscopy with MS (IR/MS) with GC/MS has been common practise as a practical technique for the separation and detection of volatile compounds (Wilkins 1994).

2. Aims of the Thesis

2.1 Aim of the study

The aim of this study is to investigate the effect of drying temperature on the chemical composition of the extract of selected spices.

2.2 Objectives of the study

The objectives of the study are:

1. Extraction and analysis of volatile substances of selected spices
2. Evaluation of the effect of different drying methods on the volatile compounds in selected spices and comparison with commercially available dried spices

3. Materials and Methods

3.1 Location of experiment

The experiment was conducted at the Food processing technologies Laboratory as well as Laboratory of ethnobotany and ethnopharmacology, Faculty of Tropical Agrisciences, Czech University of Life Science, Prague. The experiment was conducted between November 2021 to March 2022.

3.2 Experimental materials

A total of six (6) different spices were used for this experiment which include parsley, sage, basil, rosemary, ginger, and thyme. The fresh herbs were bought from a local market. Basil was grown from Kenya, rosemary from Morocco, while parsley, sage, ginger and thyme are from Czech Republic. The studied herbs were also purchased in dried form (Petržel, Šalvěj, Bazalka, Rozmarýn, Zázvor, Tymián - Kotányi, s.r.o.), these samples are marked as "commercial".

3.3 Equipment and reagents

The equipment used for the purpose of the experiment include Series Automatic Soxhlet Extractor SER 158 – Velp Scientifica, sensitive weighing balance, petri dishes, spatula, tubes, round bottom flask, mill (RETSCH Knife Mill Grindomix GM 100), drying oven (Memmert Oven UN110m plus, Merci s.r.o.), , 500 microlitres pipettes, vacuum rotary evaporator (Heidolph Hei-VAP Core rotary evaporator – VERKON s.r.o.) and Gas chromatography/Mass spectrometry analyser (Agilent Technologies 5977A MSD equipped with a 38 HP-5 column (5%-phenyl)-methylpolysiloxane, 30 m length, 250 μm internal diameter, 0.25 μm film thickness). All reagents and chemicals used were of analytical grade; they include: n-Hexane (Penta s.r.o.).

3.4 Extraction and GC/MS analysis

The methodology was modified according to the 'Effect of different drying methods on the volatile components of parsley (*Petroselinum crispum* L.) M.C. Díaz-Maroto, M.S. Pérez-Coello & M.D. Cabezudo.

GC/MS analysis was conducted by the method described by Farouk et al. (2017). The collected fresh samples were divided into two parts, and different drying methods were applied. Room temperature drying was carried out in a partly closed place protected from sunlight (average temperature was 22 °C) by using trays; the duration was 7 days. After the drying process, a constant weight was achieved. The rest of the plant material was dried in the oven in Petri dishes plates at a temperature 45 °C to constant weight (10-15 hrs, depending on the type of spice). After drying, the leaves were removed from the stems for further analysis.

A modified AOAC 2003.06 extraction was used for extraction. 7 g of the dried sample was placed in an extraction thimble with 130 ml of hexane. After extraction, the solvent was evaporated on a vacuum rotate evaporator. The sample was then diluted with hexane to a concentration of 10 µl/ml.

Each sample (commercial, room temperature, 45 °C) was analysed in triplicates. Overall, there were fifty-four measurements. GC/MS analysis was employed. The injections were performed using an autosampler immediately after extraction. Injection volume was 1 µl. The inlet GC injection port temperature was maintained at, 220 °C, the split mode was setto 1:10. The optimized GC oven temperature program was 50 °C (3 mins) to 120 °C (rate 3 °C/min) to 250 °C (rate 5 °C/min), hold time 5 min, to 280 °C (rate 15 °C/min) hold 5 min. Carrier gas helium was used at a flow rate of 1 mL/min. The MSD transfer line temperature was maintained at, 250 °C with the electron energy of 70 eV. Mass spectra were acquired in the mass range from m/z 30 to 600, using a scan time of 1 s. Data was obtained through MassHunter Workstation Software Qualitative Analysis Version B.07.00.

Percentage composition was obtained from electronic integration measurements. The representation of the individual components is presented as the average value from three repeated measurements. Identification of constituents was based on a comparison of their retention indices (RI) and spectra with the National Institute of Standards and Technology Library ver. 2.2.f (NIST, USA), as well as with authentic standards and literature (Adams 2007). The RI were calculated using the retention times of n-alkanes series ranging from C7

to C40 (Sigma-Aldrich, Prague, CZ). Not all substances could be verified by comparison of RI, because some retention indexes were not available.

4. Results

4.1 Volatile compounds of parsley EO

Table 1 shows the volatile compound composition of parsley essential oil as affected by drying method. A total of thirteen volatile compounds were identified in commercial parsley EO, twelve were identified in parsley EO air-dried at room temperature and four were identified in oven-dried parsley EO. Apiol was identified as the most abundant compound in parsley. The result revealed that commercial parsley contained 76.48 % apiol while parsley dried at room temperature and 45 °C oven-drying contained 65.45 % and 93.64 % apiol, respectively. More so, small amounts of p-Mentha-1(7),8-diene, Caryophyllene, and Phytol were found in commercial parsley, air-dried parsley and parsley oven-dried at 45 °C.

The result also revealed that α-Pinene, β-Myrcene, *o*-Cymene, Terpinolene, 1,3-Dimethyl-2-vinylbenzene, γ-Elemene, and Germacrene D were found in little proportions in parsley dried at room temperature but were not found in commercial parsley and parsley dried at 45 °C oven-drying. However, Linalool, (-)-Carvone, Anethole, α-Terpinyl acetate, α-Guaiene, 1,3-Benzodioxole 4-methoxy-6-(2-propenyl), and Caryophyllene oxide were found in little proportions in commercial parsley but were not identified in air-dried parsley and oven-dried parsley.

Table 1: Chemical composition of parsley essential oil

RT (min)	Compound	Retention Index		Relative content (%)		
		Pub.	Obs.	Comm.	R. temp.	45 °C
12.14	α-Pinene	939	920	0.00	0.75	0.00
15.02	β-Myrcene	981	978	0.00	2.61	0.00
16.74	o-Cymene	1011	1012	0.00	4.75	0.00
16.91	p-Mentha-1(7),8-diene	1004	1015	0.58	7.45	1.47
19.91	Terpinolene	1088	1073	0.00	1.35	0.00
20.11	1,3-Dimethyl-2-vinylbenzene	1074	1077	0.00	4.03	0.00
20.76	Linalool	1098	1089	0.87	0.00	0.00
27.71	(-)-Carvone	1242	1231	4.44	0.00	0.00
29.72	Anethole	1289	1275	1.22	0.00	0.00
32.00	α-Terpinyl acetate	1352	1331	1.43	0.00	0.00
34.45	Caryophyllene	1428	1399	4.31	2.93	1.92
34.84	γ-Elemene	1430	1411	0	1.01	0
36.37	Germacrene D	1480	1462	0.00	1.42	0.00
36.55	α-Guaiene	1439	1468	2.95	0.00	0.00
37.54	β-Sesquiphellandrene	1519	1500	0.51	2.49	0.00
37.97	1,3-Benzodioxole 4-methoxy-6-(2-propenyl)	1520	1516	1.15	0.00	0.00
39.28	Caryophyllene oxide	1573	1565	0.69	0.00	0.00
39.68	Carotol	1594	1580	2.38	0.00	0.00
41.76	Apiol	1680	1663	76.48	65.45	93.64
50.44	Phytol	2135	2170	2.99	5.76	2.97

RT = retention time, min = minutes, Pub = Published, Obs. = Observed, Comm. = commercial, R. temp. = room temperature.

4.2 Volatile compounds of rosemary EO

Volatile compounds identified in rosemary essential oil as affected by the drying method are presented in Table 2. A total of 14 volatile compounds were identified in commercial rosemary EO, 22 volatile compounds were identified in air-dried sample and 23 volatile compounds were identified in oven-dried sample. The analysis identified Eucalyptol as the most abundant volatile compound in commercial rosemary (47.73 %), air-dried rosemary (22.95 %) and oven-dried rosemary (19.28 %). The result also revealed that Camphor was also abundant in commercial rosemary (20.47 %), air-dried rosemary (18.15 %) and oven-dried rosemary (12.82 %). Another abundant volatile compound identified in rosemary is α-Pinene. This study recorded 9.40 % of α-Pinene in commercial rosemary, 7.41 % in air-dried rosemary and 18.25 % in oven-dried rosemary.

Furthermore, Caryophyllene, Camphene, β-Pinene, α-Terpinene, Linalool, Endo-borneol, Terpinen-4-ol, α-Terpineol, (-)-Bornyl acetate, Humulene, and Caryophyllene oxide were found in little proportions in commercial rosemary, air-dried rosemary and oven-dried rosemary. However, Tricyclene, Dehydrosabinene, β-Myrcene, α-Phellandrene, γ-Terpinene, *E*-Sabinene hydrate, Terpinolene, 2-Pinen-4-one, and Methyleugenol were found in rosemary dried at room temperature and oven-dried rosemary but absent in commercial rosemary.

Table, 2: Chemical composition of rosemary essential oil

RT	Compound	Retention Index		Relative content (%)		
		Pub.	Obs.	Comm.	R. temp.	45 °C
11.56	Tricyclene	926	909	0.00	0.00	0.27
12.15	α-Pinene	937	920	9.40	7.41	18.25
12.85	Camphene	953	934	3.01	3.69	6.44
13.17	Dehydrosabinene	960	941	0.00	0.27	0.33
14.23	β-Pinene	980	962	1.01	1.98	2.88
15.03	β-Myrcene	991	978	0.00	0.48	1.14
15.68	α-Phellandrene	1005	991	0.00	2.05	1.78
16.31	α-Terpinene	1018	1003	0.50	1.16	0.97
17.13	Eucalyptol	1033	1019	47.73	22.95	19.28
18.46	γ-Terpinene	1062	1045	0.00	1.59	1.15
19.06	*Z*-Sabinene hydrate	1069	1056	0.00	0.45	0.37
19.92	Terpinolene	1088	1073	0.00	0.74	0.59
20.76	Linalool	1112	1089	1.07	0.64	1.30
22.89	Camphor	1143	1132	20.47	18.15	12.82
24.14	endo-Borneol	1165	1156	5.22	4.33	4.55
24.55	Terpinen-4-ol	1177	1165	0.68	0.68	0.68
25.36	α-Terpineol	1189	1181	4.41	2.98	1.96
26.16	2-Pinen-4-one	1205	1197	0.00	4.98	7.08
29.44	(-)-Bornyl acetate	1285	1269	0.83	11.67	9.13
34.04	Methyleugenol	1401	1387	0.00	0.43	0.24
34.48	Caryophyllene	1428	1399	4.27	10.52	6.71
35.54	Humulene	1440	1434	0.59	1.64	1.06
39.28	Caryophyllene oxide	1581	1565	0.83	1.19	1.02

RT = retention time, min = minutes, Pub. = Published, Obs. = Observed, Comm = commercial, R. temp. = room temperature.

4.3 Volatile compounds of sage EO

The volatile compounds identified in sage essential oil as affected by the drying method are presented in Table 3. A total of 21 volatile compounds were identified in commercial sage EO, 18 volatile compounds were identified in air-dried sample and 16 volatile compounds were identified in oven-dried sample. The analysis identified 2-Bornanone, Eucalyptol and Epimanool as abundant volatile compounds in sage. The study found 21.99 % 2-Bornanone in commercial sage, 18.04 % in air-dried sage and 27.02 % in oven-dried sage. The result also identified 9.94 % Eucalyptol in commercial sage, 20.55 % in air-dried sage and 20.24 % in oven-dried sage. The result also shows that commercial sage contained 9.41 % Epimanool, air-dried sage contained 14.50 %, and oven-dried sage contained 28.42 %.

Furthermore, Camphene, Terpinolene, γ-Terpinene, β-Thujone, Endo-borneol, L-α-Terpineol, L-α-Bornyl acetate, (+)-Ledene and Viridiflorol were found in small proportions in commercial sage, air-dried sage and oven-dried sage. However, 2-Thujene, β-Pinene, β-Myrcene, and *E*-Sabinene hydrate were found in sage dried at room temperature and oven-dried sage but absent in commercial sage.

Table 3: Chemical composition of sage essential oil

RT	Compound	Retention Index		Relative content %		
		Pub.	Obs.	Comm.	R. temp.	45 °C
11.98	2-Thujene	931	917	0.00	1.56	0.85
12.24	α-Pinene	937	922	2.07	2.47	0.00
12.87	Camphene	953	935	2.62	3.88	2.93
14.27	β-Pinene	980	963	0.00	4.44	2.79
15.23	β-Myrcene	991	982	0.00	5.99	2.43
16.46	Terpinolene	1088	1006	0.52	1.17	1.12
17.04	Eucalyptol	1033	1017	9.94	20.55	20.24
18.47	γ-Terpinene	1062	1045	0.30	1.71	1.56
19.26	*E*-Sabinene hydrate	1070	1060	0.00	0.00	0.43
21.48	β-Thujone	1102	1103	2.92	6.33	6.48
23.00	2-Bornanone	1145	1134	21.99	18.04	27.02
24.18	endo-Borneol	1165	1157	4.02	0.93	1.41
24.58	Terpinen-4-ol	1177	1165	0.40	0.00	0.00
25.34	L-α-Terpineol	1189	1180	0.35	0.45	0.50
27.72	Carvone	1242	1231	0.36	0.00	0.00
29.51	L-α-bornyl acetate	1285	1270	4.24	1.14	1.56
29.78	Thujyl acetate	1290	1276	0.55	0.00	0.00
34.55	Caryophyllene	1428	1402	6.45	7.18	0.00
35.67	Humulene	1440	1439	10.79	0.00	1.54
36.65	β-Eudesmene	1485	1471	0.00	0.00	0.00
36.81	(+)-Ledene	1490	1476	2.04	1.67	0.30
39.3	Caryophyllene oxide	1581	1566	0.58	0.00	0.00
39.75	Viridiflorol	1590	1583	17.34	7.44	0.40
40.04	Humulene epoxide	1607	1593	1.61	0.00	0.00
40.07	Epiglobulol	1588	1594	0.00	0.53	0.00
41.28	Isoaromadendrene epoxide	1612	1643	1.50	0.00	0.00
49.9	Epimanool	2056	2142	9.41	14.50	28.42

RT = retention time, min = minutes, Pub. = Published, Obs. = Observed, Comm = commercial, R. temp. = room temperature.

4.4 Volatile compounds of ginger EO

Table 4 presents the volatile compounds of essential oil identified in ginger as affected by the drying method. This study identifies a total of 22 volatile compounds in commercial ginger EO, 22 volatile compounds were identified in air-dried sample and 27 volatile compounds were identified in oven-dried sample. The analysis identified Curcumene, β-Bisabolene, and β-Sesquiphellandrene as abundant volatile compounds in commercial and air-dried ginger. The study found 37.25 % Curcumene in commercial ginger, 11.01 % in air-dried ginger and 27.02 % in oven-dried ginger. The result also identified 10.06 % β-Bisabolene in commercial ginger, 21.55 % in air-dried ginger and 0.79 % in oven-dried ginger. The result also shows that commercial ginger contained 26.31 % β-Sesquiphellandrene, air-dried ginger contained 20.05 % and oven-dried ginger contained 1.00 %. The study found β-Elemene, Epi-β-Caryophyllene, and alfa-Copaene as abundant volatile compounds in oven-dried ginger. The result showed that oven-dried ginger contained 17.16 % β-Elemene, 14.07 % Epi-β-Caryophyllene, and 11.51 % α -Copaene.

Furthermore, α-Pinene, Camphene, β-Myrcene, Decane, Octanal, *Z*-Sabinene hydrate were absent in commercial ginger and air-dried ginger, but they were available in small proportions in oven-dried ginger. However, Eucalyptol, Melonal, Linalool, 6-Octenal, endo-Borneol, α-Terpineol, Decanal, β-Citral, Geraniol, α-Citral, 2-Undecanone, and Cyclosativene were found in commercial ginger, air-dried ginger at room temperature and oven-dried ginger.

Table 4: Chemical composition of ginger essential oil

RT	Compound	Retention Index		Relative content (%)		
		Pub.	Obs.	Comm.	R. temp.	45 °C
12.08	α-Pinene	920	919	0.00	0.00	1.46
12.78	Camphene	934	933	0.00	0.00	2.85
14.98	β-Myrcene	981	977	0.00	0.00	1.94
15.35	Decane	999	984	0.00	0.00	0.56
15.62	Octanal	1001	990	0.00	0.00	1.02
16.87	*Z*-Sabinene hydrate	1089	1014	0.00	0.00	3.25
16.9	Eucalyptol	1019	1015	1.85	4.66	4.24
18.18	Melonal	1056	1039	0.46	0.00	0.42
20.69	Linalool	1089	1088	1.25	0.88	0.47
23.16	6-Octenal	1153	1137	0.00	0.57	1.15
24.05	endo-Borneol	1156	1155	2.54	1.02	2.33
25.26	α-Terpineol	1189	1179	1.58	1.14	4.18
25.72	Decanal	1204	1188	1.10	0.59	2.33
27.59	β-Citral	1218	1228	0.00	7.43	1.76
28.25	Geraniol	1255	1242	0.64	0.00	0.57
29.12	α-Citral	1271	1262	0.00	17.46	4.37
29.79	2-Undecanone	1291	1276	0.00	0.64	0.64
32.55	Cyclosativene	1368	1346	0.55	0.36	3.20
32.91	α -Copaene	1376	1356	1.22	1.11	11.51
33.49	β-Elemene	1375	1372	1.62	1.79	17.16
33.88	7-epi-Sesquithujene	0	1383	0.75	0.00	3.12
34.39	epi-β-Caryophyllene	0	1397	0.74	0.74	14.07
34.83	Elixene	1445	1411	1.59	1.67	0.81
35.5	*E*-β-Farnesene	1458	1433	2.17	1.26	4.14
36.21	γ-Muurolene	1477	1456	1.89	1.86	3.01
36.51	Curcumene	1486	1466	37.25	11.01	2.47
37.21	β-Bisabolene	1509	1489	10.06	21.55	0.79
37.7	β-Sesquiphellandrene	1519	1506	26.31	20.05	1.00
38.39	Elemol	1547	1532	1.34	1.41	1.86
38.61	*E*-Nerolidol	1564	1540	1.46	0.85	1.12
39.98	Zingiberenol	1626	1591	1.68	0.81	1.66
40.46	Globulol	1576	1610	1.96	1.14	0.56

RT = retention time, min = minutes, Pub. = Published, Obs. = Observed, Comm. = commercial, R. temp. = room temperature.

4.5 Volatile compounds of basil EO

The volatile compounds identified in basil essential oil as affected by the drying method are presented in Table 5. This study identifies a total of 24 volatile compounds were identified in commercial basil EO, 15 volatile compounds were identified in air-dried sample and 22 volatile compounds were identified in oven-dried sample. The result identified Eugenol, α-Bergamotene, and Linalool as abundant volatile compounds in commercial, air-dried, and oven-dried basil. The study found 3.77 % Eugenol in commercial basil, 59.78 % in air-dried basil and 37.35 % in oven-dried basil. The result also identified 10.16 % α-Bergamotene in commercial basil, 12.51 % in air-dried basil and 16.50 % in oven-dried basil. The result also shows that commercial basil contained 10.28 % Linalool, air-dried basil contained 6.38 %, and oven-dried basil contained 16.36 %. The study found Estragole (16.24 %), and 2-Propenoic acid 3-phenyl- methyl ester (25.43 %) as abundant volatile compounds in commercial basil.

4.6 Volatile compounds of thyme EO

Table 6 shows the volatile compound composition of Thyme EO as affected by the drying method. A total of 9 volatile compounds were identified in commercial thymus EO, 11 volatile compounds were identified in air-dried sample and 13 volatile compounds were identified in oven-dried sample. Thymol, γ-Terpinene and m-Cymene were identified as the abundant compounds in Thyme. The result revealed that commercial thyme contained 71.01 % Thymol while thyme dried at room temperature and 45 °C oven-drying contained 58.70 % and 59.98 % Thymol, respectively. The result also showed that 18.55 % γ-Terpinene was contained in commercial thyme while air-dried commercial thyme contained 16.65 % and oven-dried commercial thyme contained 14.59 %. Similarly, commercial thyme contained, 2.88 % m-Cymene while air-dried thyme contained 13.14 % and oven-dried thyme contained 11.74 %.

Furthermore, small amounts of *E*-Sabinene hydrate, Linalool, Endo-Borneol, Methyl thymyl ether, Methyl carvacrol and Caryophyllene were found in commercial, air-dried and Thyme oven-dried at 45 °C. The result also revealed that, 2-Thujene and Terpinolene were found in small proportions in thyme dried at room temperature and thyme oven-dried at 45 °C but

were not found in commercial thyme. α-Pinene and β-Myrcene were found in minor amont in thyme oven-dried at 45 °C but were not identified in air-dried thyme and commercial thyme.

Table 5: Chemical composition of basil essential oil

RT	Compound	Retention Index		Relative content (%)		
		Pub.	Obs.	Comm.	R. temp.	45 °C
17.04	Eucalyptol	1033	1017	0.63	1.15	2.17
20.9	Linalool	1098	1092	10.28	6.38	16.36
24.23	Isoborneol	1156	1158	0.30	0.00	0.24
25.36	L-α-Terpineol	1189	1181	0.00	0.68	1.41
25.41	Terpinen-4-ol	1182	1182	0.78	0.00	0.00
25.69	Estragole	1195	1187	16.24	0.00	0.00
29.51	Bornyl acetate	1285	1270	0.54	0.82	1.35
29.77	Anethole	1289	1276	0.45	0.00	0.00
31.51	γ-Elemene	1433	1433	0.00	0.91	0.58
32.74	Eugenol	1356	1352	3.77	59.78	37.35
33.56	β-Elemen	1375	1374	0.00	2.54	2.76
34.16	Methyleugenol	1401	1391	7.74	0.00	0.27
34.44	β-Ylangene	1421	1398	0.00	0.65	0.91
34.51	Caryophyllene	1418	1400	2.05	0.00	0.28
34.98	α-Bergamotene	1436	1416	10.16	12.51	16.50
35.53	*E*-β-Farnesene	1458	1434	0.72	3.03	3.47
35.6	Humulene	1440	1436	0.38	0.00	0.80
35.88	*E*-Muurola-4(15),5-diene	1463	1446	0.78	0.86	0.76
36.44	Isogermacrene D	1448	1464	1.81	3.53	4.02
36.6	β-Eudesmene	1472	1469	0.67	0.00	0.00
36.84	Bicyclogermacren	1494	1477	0.00	0.98	1.46
36.85	γ-Eudesmol	1472	1478	0.82	0.00	0.00
37.02	α-Bulnesene	1505	1485	0.00	0.00	1.46
37.41	γ-Cadinene	1513	1496	6.07	3.57	4.26
38.67	1,6,10-Dodecatrien-3-ol-3,7,11-trimethyl-	1566	1542	0.29	0.00	0.00
39.27	Spathulenol	1575	1565	1.08	0.00	0.75
39.34	Caryophyllene oxide	1575	1567	0.72	0.00	0.00
40.03	Humulene epoxide, 2	1607	1593	0.56	0.00	0.00
50.47	Phytol	2114	2171	3.85	2.59	2.48

RT = retention time, min = minutes, Pub. = Published, Obs. = Observed, Comm. = commercial, R. temp. = room temperature.

Table 6: Chemical composition of thyme essential oil

		Retention Index		Relative content (%)		
RT	Compound	Pub.	Obs.	Comm.	R. temp.	45 °C
11.8	2-Thujene	931	913	0.00	1.14	1.56
12.13	α-Pinene	920	920	0.00	0.00	0.76
15.01	β-Myrcene	981	977	0.00	0.00	1.89
16.25	Terpinolene	1088	1002	0.00	1.93	2.04
16.69	m-Cymene	1023	1011	2.88	13.14	11.74
18.52	γ-Terpinene	1045	1046	18.55	16.65	14.59
19.00	*E*-Sabinene hydrate	1056	1055	1.19	1.15	0.95
20.69	Linalool	1089	1088	1.19	2.25	1.49
24.08	endo-Borneol	1156	1155	0.72	0.81	1.33
27.12	Methyl thymyl ether	1232	1218	1.04	0.95	0.60
27.56	Methyl carvacrol	1244	1227	1.07	0.97	0.76
30.87	Thymol	1290	1300	71.01	58.70	59.98
34.44	Caryophyllene	1428	1398	2.34	2.32	2.30

RT = retention time, min = minutes, Pub. = Published, Obs. = Observed, Comm. = commercial, R. temp. = room temperature.

5 Discussion

This study identified apiol as the most abundant volatile compound in parsley, whereby the highest relative content of apiol was recorded in extract oven-dried at 45 °C, which was greater than apiol content recorded in air-dried parsley and commercial parsley. According to Zhang et al. (2006), antioxidant activity in parsley essential oil is due to apiol, which is described as the major contributor to the antioxidant activity of this oil. Apiol has also been explored as a conceivable abortifacient. Hence, pregnant women ought to be mindful so as not to eat a lot of parsley (Ajmera et al. 2019). The result also showed that drying method affected the quantity of volatile compounds recorded in parsley, especially apiol, in favour of oven-drying at 45 °C.

However, the relative content of other more volatile compounds decreased at the expense of the higher content of Apiol during oven-drying at 45 °C compared to drying at room temperature. This means that oven-drying at 45 °C negatively affected the chemical composition of the essential oil. These results are in agreement with those obtained by Badee et al. (2020), who reported that oven-drying at 45 °C caused the greatest losses in the volatiles. Kandil et al. (2016) mentioned that the oil content of dried parsley was strongly affected by drying methods. These findings are similar to Díaz-Maroto et al. (2002), who also found apiol to be the most abundant volatile compound in parsley. Macleod et al. (1985) also reported 45 volatile compounds from parsley leaves and identified apiol as one of the most abundant volatile components of parsley.

This study identified Eucalyptol, Camphor and α-Pinene as the abundant volatile compounds in rosemary. Oven-drying of rosemary at 45 °C caused a great reduction in the proportion of Eucalyptol and Camphor but however increased the proportion of α-Pinene in rosemary EO. Anh et al. (2019) also reported Eucalyptol, Camphor and α-Pinene as abundant compounds in rosemary essential oil in Vietnam. Eucalyptol, 2-Bornanone and Epimanool were identified as abundant volatile compounds in sage by this study. Oven-drying at 45 °C did not reduce the proportions of these compounds compared to air-drying at room temperature. However, some compounds such as α-Pinene, Terpinen-4-ol and Caryophyllene disappeared during oven-drying. The results of the tests of essential oil content conducted on sage by

Sellami et al. (2012) indicated losses from 0.3 % to 0.26 % during convection-drying method at 45 °C compared to naturally drying at approximately 22 °C, which is comparable to the values presented in this study. Pirbalouti et al. (2013) observed the lowest oil losses in green and red basil dried naturally in the shade. This method of conservation, though the least expensive, is linked to the risk of adverse atmospheric conditions and prolonged time of drying, during which enzymatic decomposition and the development of unwanted microbiota may occur. Eucalyptol, Camphor and α-Pinene have been reported to possess antimicrobial activity against a range of bacteria such as *E. coli*, *S. aureus* and *Bacillus* species (Zengin & Baysal 2014).

The result of this study reveals that drying ginger at 45 °C increased the concentration of α-Copaene (11.51 %), β-Elemene (17.16 %) and Epi-β-Caryophyllene (14.07 %) compared to drying at room temperature and commercial ginger. Oven-drying of ginger at 45 °C also favoured the availability of α-Pinene, Camphene, β-Myrcene, Decane, Octanal and *Z*-Sabinene hydrate that were absent in ginger air-dried at room temperature. However, oven-drying lead to the decrease of β-Bisabolene, Curcumene, and β-Sesquiphellandrene which have been reported to be dominant compounds in ginger. The EOs of ginger rhizomes are used for preserving various foods against autoxidation and microbial spoilage because of their antioxidant and antimicrobial properties (Bellik 2014). Many in vitro studies demonstrated the antimicrobial potential of *Zingiber* plant extracts against both Gram-positive (*Bacillus cereus*, *Staphylococcus aureus*) and Gram-negative (*Escherichia coli*, *Salmonella typhi*, *Pseudomonas aeruginosa*, *Klebsiella pneumonia*) bacteria (Kumar et al. 2011). The EOs also exhibited significant antifungal activity against *Candida glabrata*, *C. albicans* and *Aspergillus niger* (Ghosh et al. 2011). These results suggest that EO of *Zingiber* plant could be used in the treatment of many bacterial and fungal diseases as well as in food preservation as natural preservatives (Bellik 2014). These results of this study are in agreement with the findings of Huang et al. (2012), who studied the effects of oven-drying, microwave drying, and silica gel drying methods on the degree of dehydration and volatile components of ginger. Sixty compounds were identified by GC/MS.

This study found increasing abundanceof Linalool (16.36 %) and α-Bergamotene (16.50 %) in oven-dried basil compared to air-dried basil and commercial basil. Meanwhile, air-drying of basil at room temperature resulted in higher retention of Eugenol (59.78 %), which was lost in oven-dried basil. Linalool has a variety of biological properties, including anxiolytic, sedative, anti-inflammatory, anticonvulsant, and analgesic effects (Aprotosoaie et al. 2014). The results obtained from the present experiment and reports of other scientists show that there was a contradictory viewpoint on the effects of different drying methods on the essential oil profile of basil. Drying conditions meaningfully influenced the essential oil composition of *Calendula officinalis,* and the predominant constituents of the oil were monoterpenoid components (Okoh et al. 2008). Furthermore, consistent with this study, oven-drying led to the destruction and/or absence of some major components (Okoh et al. 2008). Contrarily, Sefidkon et al. (2006) reported that drying methods had no significant effect on essential oil composition of spices.

This study shows that there is no significant difference in thyme dried at room temperature and 45 °C. However, during oven-drying, essential compound such as γ-Terpinene was lost. γ-Terpinene has been reported by several researchers to possess antimicrobial, antifungal and antiviral properties (Carson & Riley 2014). Similarly, Calín-Sánchez et al. (2013) evaluated the effects of different methodologies and temperatures in the chemical composition of thyme essential oils, which presented high levels of thymol after oven-drying. The authors found that oven-drying promoted an increase in the levels of thymol, which corroborate with the results of this study.

6. Conclusions

This study explored the effect of drying methods on volatile compounds of six spices, namely parsley, rosemary, sage, ginger, basil and thyme. The study concludes that apiol is the most abundant volatile compound in parsley and oven-drying at 45 °C increased the availability of apiol in parsley. However, most of the parsley essential oil components decreased during oven-drying at 45 °C compared to room temperature drying. This study also identified Eucalyptol, Camphor and α-Pinene as the abundant volatile compounds in rosemary. Oven-drying of rosemary at 45 °C caused a great reduction in the proportion of Eucalyptol and Camphor but increased the relative content of α-Pinene in rosemary.

Furthermore, oven-drying at 45 °C did not affect the presence of Eucalyptol, 2-Bornanone and Epimanol in the sage extract compared to air-drying at room temperature. Oven-drying of ginger at 45 °C also favoured the availability of α-Pinene, Camphene, β-Myrcene, Decane, Octanal and *Z*-Sabinene hydrate that were absent in ginger air-dried at room temperature. However, oven-drying lead to the decrease of β-Bisabolene, Curcumene, and β-Sesquiphellandrene which have been reported to be dominant compounds in ginger. Oven-dried basil also contained higher concentration of Linalool (16.36 %) and α-Bergamotene compared to air-dried samples.

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