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Extraction and Volatile Compounds in Ginger Essential Oil (*Zingiber officinale* Roscoe) at Laboratory Scale

CHI BICH THAO[1], THI TUU TRAN[2,3], THI KIM NGAN TRAN[2,3,*] and HUYNH CANG MAI[1,*]

[1]Faculty of Chemical Engineering and Food Technology, Nong Lam University, Ho Chi Minh City 700000, Vietnam
[2]Institute of Applied Technology and Sustainable Development, Nguyen Tat Thanh University, Ho Chi Minh City, Vietnam
[3]Faculty of Environmental and Food Engineering, Nguyen Tat Thanh University, Ho Chi Minh City, 700000, Vietnam

*Corresponding authors: Tel: +84 938 228017; E-mail: nganttk@ntt.edu.vn; maihuynhcang@hcmuaf.edu.vn

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Ginger (*Zingiber officinale* Roscoe) has a characteristic pungent taste and contains biologically active substances with many potential benefits for use in many different fields. Ginger essential oil is extracted from ginger root (*Zingiber officinale*) through steam distillation. The main compound in the essential oil was determined by the gas chromatography-mass spectrometry method and evaluated antioxidant activity by measuring DPPH. Essential oil of ginger yields 2.65 mL/g and comprises the primary chemical constituents such as zingiberene (18.95 %) which is the fragrance ingredient for the characteristic scent of ginger and some other ingredients camphene, β-phellandrene, α-citral, eucalyptol, sesquiphellandrene, α-farnesene, α-pinene, *etc.* The antioxidant activity of ginger essential oil was evaluated by the DPPH (2,2-diphenyl-1-picrylhydrazyl) method with radical scavenging ability and oxidation reached 74.19%. The present work was carried out for the purpose of developing products and applying ginger essential oil in the fields of pharmaceutical, cosmetic and food industries, clarifying the advantages and disadvantages and improving the value of ginger in different regions locally.

Keywords: *Zingiber officinale*, Essential oil, Hydrodistillation, GC-MS, DPPH method.

INTRODUCTION

Ginger (*Zingiber officinale* Roscoe) is a herbaceous perennial, native to Southeast Asia and has been cultivated in tropical and subtropical regions around the world for centuries [1]. Ginger can only flourish in climates that are both warm and humid. Although ginger plants can tolerate some direct sunlight, they generally prefer partial shade or filtered light. This is the reason why ginger is frequently cultivated behind larger trees or in shaded areas, away from direct exposure to sunshine [2-5]. Ginger is usually harvested 8 to 10 months after planting when the leaves begin to turn yellow. Some of the countries that grow the most ginger in the world such as India, China, Indonesia, Nepal, Thailand, Nigeria, *etc*.. Ginger cultivation in Vietnam exhibits a wide geographical distribution, encompassing both domestic gardens and other containers such as barrels. However, it is crucial to ensure that the requisite growing conditions are adequately fulfilled [6].

Ginger has many benefits and uses to help reduce inflammation, improve digestion, protect and fight skin aging, deal with dandruff, stimulate hair growth and aromatherapy, *etc*. Moreover, it is important to remember that ginger essential oil has a quite characteristic pleasant smell. Ginger essential oil is extracted from the rhizome (root) of the ginger plant by various methods. However, there are several other methods that can be used to extract essential oils including steam distillation, hydrodistillation, cold press extraction, supercritical fluid extraction and solvent extraction, among which the most common and traditional method for extraction of ginger essential oil is steam distilled [7-10]. The choice of extraction method can affect the quality, aroma and composition of the extracted essential oil.

A complex blend of aromatic components gives ginger essential oil its signature scent, flavour and pharmacological potential. The composition of ginger essential oil can vary depending on the several factors such as source of ginger plant, growing conditions and the extraction method used. Some of the main components commonly found in a ginger essential oil that contribute to the spicy aroma are zingiberene, gingerol,

zingerone and scent components such as β-sesquiphellandrene, α-pinene, β-pinene, camphene and phellandrene [11-13]. The combination of these compounds gives ginger essential oil its unique aroma and potential therapeutic benefits. Previous studies have demonstrated compounds in ginger essential oil to be effective against a variety of bacteria, including some pathogenic strains. In addition, gingerol, which is the main bioactive compound in fresh ginger and ginger essential oil, has been shown to have antioxidant and anti-inflammatory properties, helps scavenge free radicals and may contribute to the overall antioxidant capacity of ginger essential oil [14-16].

In Vietnam, ginger is a crop whose prices fluctuate widely depending on several factors. Including when to buy seeds, farming methods, varieties and markets, so the price of ginger seeds will change continuously from time to time, directly affecting farmers. Therefore, in order to improve the economic value and agricultural products of farmers, taking advantage of abundant raw materials, the selection of essential oil extraction research is a promising direction. The study conducted the extraction of ginger essential oil at a laboratory scale with the hydrodistillation method, based on the influence of factors of time, solvent ratio, temperature and material size. Since then, essential oils have been determined for antioxidant activity and chemical composition by the gas chromatography-mass spectrometry (GC-MS) method.

EXPERIMENTAL

The ginger samples were procured from the Thu Duc wholesale market. The selection process involved choosing ginger roots that were three months old, ensuring freshness and absence of any physical damage or signs of insect infestation. Washed directly with clean water to remove impurities as well as dirt and then allowed the ingredients to dry naturally at room temperature.

Extraction process: Approximately 200 g of components should be cut to a thickness of approximately 1.5 cm. The cut ingredients should then be evenly distributed and subjected to a drying process at 75 ºC for specific durations of time, namely 60, 120 and 180 min. The raw materials were then ground to increase the surface area and facilitate the release of essential oils during distillation. Then, the raw materials were poured into the flask with the ratio of ingredients:water (1:1, 1:2, 1:3) and then the mixture was distilled for 3 h and sampled according to fixed timelines. The resulting condensate and essential oil mixture was anhydrous with Na_2SO_4 salt to remove water in the essential oil. Following the dehydration process, pure essential oil was calculated, subsequently preserved in Amber glass containers and stored at 4 ºC.

Method of determination of moisture: Dried and weighed the petri dish first, then weighed the sample to determine the moisture content. It was then dried to constant weight at 75 ºC. The mass of the sample lost is the amount of water present in the sample, which calculates the moisture content of the sample. Humidity was calculated as a percentage according to the following formula:

$$\text{Humidity} = \frac{m_1 - m_2}{m_1 - m_0} \times 100$$

where m_0 is the mass of the dish (g); m_1 is the mass of sample before drying (g); m_2 is the mass of sample after drying (g).

Determination of ash: The ash content was determined by heating at 600 ºC until the sample turned to white ash. Sample (2 g) was used for calcination and the ash value was calculated as follows:

$$\text{Ash degree} = \frac{G_1 - G_2}{G_1} \times 100$$

where G_1 is the mass (g) of the crucible and sample before heating and G_2 is the mass (g) of the crucible and sample after heating.

Gas chromatography-mass spectrometry (GC-MS) analysis: The sample of essential oil obtained in the best conditions will be evaluated for its chemical composition, thereby determining the content of compounds present in the pesticides remaining in the ginger essential oil. The instrument used was a GC Agilent 6890 N in combination with an HP5-MS column and an inert MS 5973 having head column pressure was 9.3 psi. Around 25 mL essential oils was added with 1 mL of *n*-hexane and dehydrated with Na_2SO_4. The flow rate was constant at 1 mL/min. The nozzle temperature was 250 ºC and the division rate is 30. Heat program for samples at 50 ºC was held for 2 min increments of 2 ºC/min to 80 ºC, continues to increase 5 ºC/min to 150 ºC continued to increase 10 ºC/min to 200 ºC and then increased further 20 ºC/min to 300 ºC hold for 5 min.

Antioxidant activity: The antioxidant activity of ginger essential oil was evaluated by DPPH (2,2-diphenyl-1-picrylhydrazyl) method. There exists an inverse relationship between the optical density (OD) value and the DPPH free radical scavenging capacity, whereby a decrease in the OD value corresponds to an increase in the scavenging capacity of DPPH free radicals. The reaction was carried out with 0.5 mL of sample (diluted DMSO) of different concentrations, then added to each test tube 3 mL ethanol and 1 mL 0.5 mM DPPH and mixed. The test tubes were kept stable in dark, at room temperature for 30 min, then the absorbance was measured at λ = 517 nm. The positive control was vitamin C, which was taken similarly. The OD value reflects the oxidation resistance of the sample. The oxidation resistance was calculated according to the formula:

$$\text{Antioxidant activity (\%)} = 100 - \left(\frac{OD_{sample} - OD_{blank}}{OD_{control}} \right) \times 100$$

RESULTS AND DISCUSSION

Ginger essential oil was recovered based on the investigation of the influencing factors of the distillation of essential oils on an experimental scale. The moisture and ash of ginger were used as an indicator for evaluating raw material quality, and the findings showed that both were reasonably high, with an average of 87.275% and an average of 6.33%, respectively. The determination of moisture and ash in fresh ingredients helps control quality and ensures the stability of the final product. These indicators can also provide useful information for determining the appropriate level of preservation and storage of materials.

Effect of drying time of raw materials on essential oil content: Based on Fig. 1, the drying time of the raw materials has an effect on the essential oil content. The most essential oil content was obtained when drying for 60 min (raw material reduced from 5-7 g). When the essential oil is not subjected to the drying process, the ginger retains a significant percentage of water content, which hinders the release of the essential oil. Consequently, the yield of essential oil obtained is lowered. When the duration of drying exceeded 60 minutes (with a reduction in raw material quantity from 12-25 g), there was a decrease in the content of essential oil. This decrease can be attributed to the volatility of essential oil during the drying process, leading to the loss of energy and subsequent release of essential oil. The content of essential oils is affected by the drying time of the raw materials and the drying process is essential in the extraction of essential oils because it affects the storage process and the concentration of volatile compounds. Prolonged drying time or exposure to excessive heat during drying can lead to deterioration of essential oils. Some heat-sensitive compounds can break down or evaporate, reducing the overall essential oil content of the material. On the other hand, if the drying process is too short or insufficient, the essential oil content may not be adequately preserved in the plant material. Rapid drying can lead to insufficient diffusion of essential oils to the plant's surface, making extraction difficult. To achieve the best results in preserving essential oil content during drying, factors such as temperature, humidity and proper air circulation should be considered, ensuring that drying conditions are controlled. Control can help maintain the quality and quantity of essential oils in the final product.

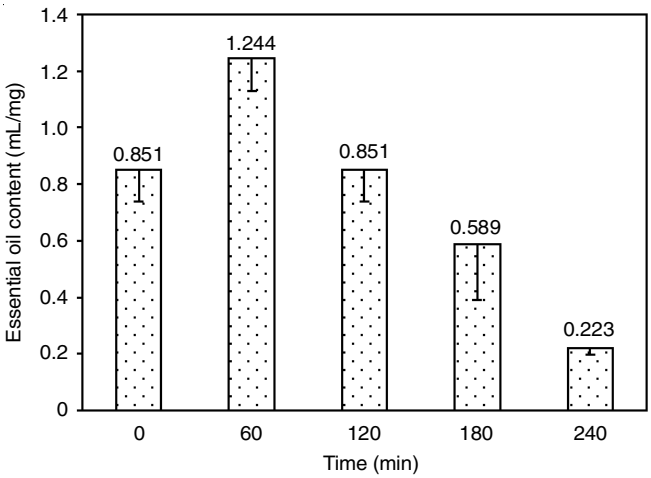

Fig. 1. Effect of material drying time

Effect of raw material size on essential oil content: Raw material size can affect essential oil content during extraction. Smaller raw material size means a larger surface area relative to its volume. When the raw material was chopped or ground into smaller pieces, it creates more surface area for extraction. This increased surface area allows for better contact between plant material and water, essential oil compounds can be released more efficiently from plant cells, resulting in a higher yield of essential oils. When using raw materials of the same size, it will ensure a more uniform extraction process, if the size changes, the extraction process will be uneven and some parts of the material will secrete more essential oil than others. other parts, resulting in significantly reduced efficiency. When the mixture was reduced to a puree (semi-solid), the oil yield was at its maximum (Fig. 2). Besides, the puree breaks the essential oil bags, permitting the essential oil to release more.

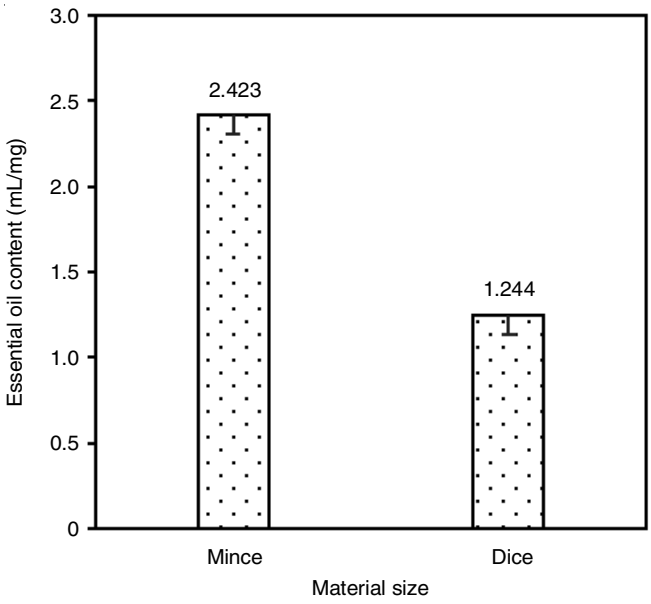

Fig. 2. Effect of material size

Effect of raw material:water ratio: Fig. 3 shows the influence of raw material and water ratio on the obtained essential oil content. Specifically, the content of essential oil obtained was highest at the ratio of 1:1 and gradually decreased later. Due to the large amount of solvent, the accumulation temperature is high, resulting in an inefficient condensing system and a small amount of essential oil is lost. If the material-water ratio is too high, the water may become saturated with the essential oil and further increase of this ratio may not result in

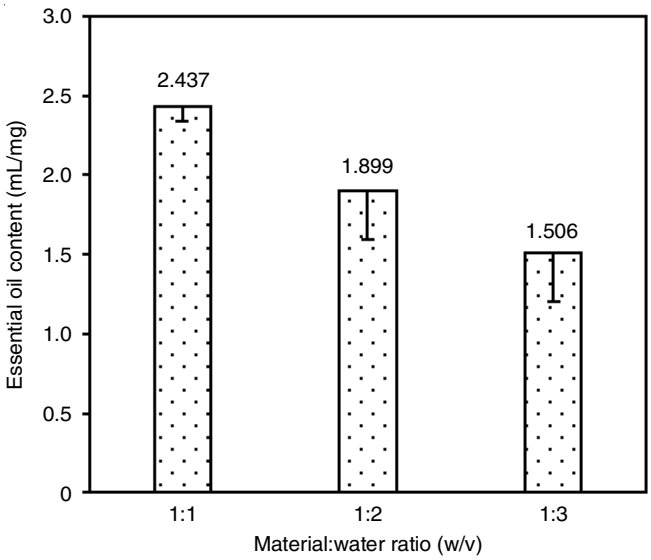

Fig. 3. Effect of material:water ratio (w/v)

a proportional increase in the essential oil content. In such cases, excess essential oils can be trapped in the plant material, reducing overall yield. Conversely, if this ratio is too low, water may not penetrate the plant material effectively, resulting in incomplete extraction of essential oil compounds.

Effect of distillation time: Distillation time can significantly affect the essential oil content of the distillation process. Based on Fig. 4, the content of essential oil increases when distillation time increased from 60 to 90 min but gradually decreases later. In the early stages of distillation, lighter and more volatile compounds tend to be collected first. Over time, the composition of essential oils can change as different compounds are released from the plant material. In some cases, prolonged distillation can extract more complex or less abundant compounds, altering the aroma and therapeutic properties of the final essential oil. However, too short distillation times can lead to incomplete extraction, while too long distillation can reduce the quality of some essential oil compounds or increase the likelihood of undesired compounds being extracted. The longer distillation time requires more energy to heat the distillation equipment and the plant material. Increasing the distillation time without proportionally increasing the yield of essential oils, which can lead to inefficient energy use and higher production costs.

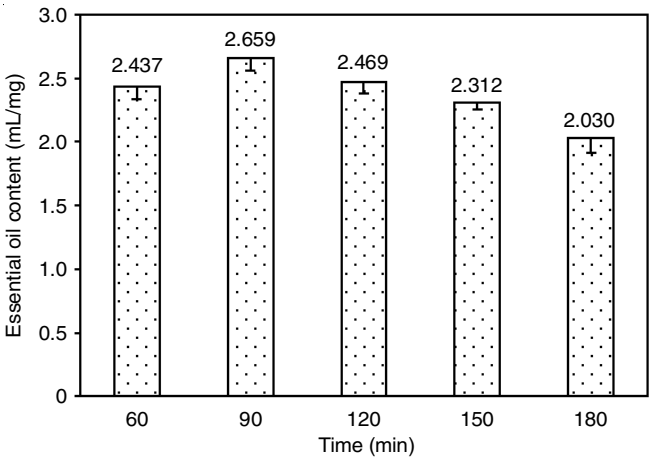

Fig. 4. Effect of hydrodistillation time

Chemical composition of ginger essential oil: Using GC-MS analysis, the volatile components in ginger essential oil were identified (Fig. 5). The results of chemical composition and content analysis are shown in Table-1. A total of 27 compounds were found, accounting for 98.92% of the total essential oil content, in which zingiberene compounds account for the highest content at 18.95% followed by components camphene (15.265%), (β-phellandrene 9.853%), α-citral (6.049%), eucalyptol (7.418%), sesquiphellandrene (6.38%), α-farnesene (5.768%), α-pinene (3.427%). The composition of essential oils can be affected by various conditions throughout the life of the plant, including growth, harvesting, postharvest handling and extraction.

Plant growth stage and harvest time can affect the concentration of essential oil components. Certain plant species may exhibit variations in oil content that correspond to distinct periods

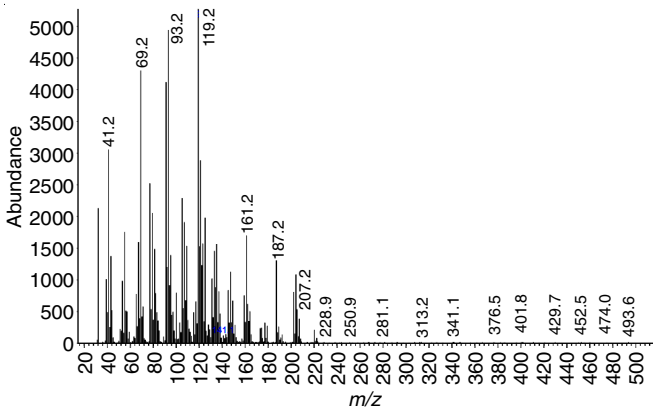

Fig. 5. Chromatography of ginger essential oil

TABLE-1
VOLATILE COMPOUNDS OF GINGER ESSENTIAL OIL

No.	RT	Compounds	Contents (%)
1	7.896	α-Pinene	3.427
2	8.520	Camphene	15.265
3	9.821	β-Pinene	0.5
4	10.691	β-Myrcene	1.606
5	11.279	α-Phellandrene	0.727
6	12.626	β-Phellandrene	9.853
7	12.734	Eucalyptol	7.418
8	17.075	Linalool	0.484
9	20.457	Borneol	1.439
10	21.674	α-Terpineol	0.732
11	23.373	Citronellol	0.808
12	23.826	β-Citral	6.049
13	24.369	*trans*-Geranio	1.796
14	24.966	α-Citral	8.93
15	25.475	Bornyl acetate	0.534
16	25.787	2-Undecanone	0.565
17	28.700	Geranyl acetate	1.292
18	28.950	β-Elemen	0.538
19	31.541	Germacrene D and α-curcumen	2.989
20	31.881	Zingiberene	18.95
21	31.941	γ-Cadinene	1.117
22	32.162	α-Farnesene	5.768
23	32.537	Sesquiphellandrene	6.38
24	33.088	Elemol	0.552
25	33.338	*trans*-Nerolidol	0.351
26	33.882	*trans*-Sesquisabinene hydrate	0.32
27	34.316	Zingiberenol	0.53

or seasons of growth. Essential oils from the same plant species but grown in different regions may exhibit differences in composition due to environmental differences. Specifically, the ginger essential oil obtained from raw materials in the Ghaziabad area of India has a relatively high compound content with sesquiterpenes (66.66%) and monoterpenes (17.28%), in which zingiberene makes up 46.71% [17]. However, the ar-curcumin component (22.1%) found in the ginger sample collected from Cuba was highest in the essential oil [18]. On the other hand, 92.7% of compounds were found in ginger essential oil in Gorakhpur (India), geranial (25.9%) was the main component in the essential oil [19]. El-Baroty *et al.* [20] reported a study on ginger root in Egypt, the essential oil extracted by hydrodistillation method with volatile compounds such as zingiberene

(13.97%), caryophyllene (15.29%) and β-sesquiphellandrene (27.16%) were the main components that account for the highest concentration in essential oils.

Radical scavenging capacity (DPPH) of ginger essential oil: Ginger essential oil has remarkable antioxidant properties due to the presence of various bioactive compounds. Some of the main components responsible for its antioxidant activity are zingiberene, camphene, eucalyptol and other phenolic compounds. From Table-2, it can be observed that there is a gradual increase in the percentage of DPPH radical scavenging activity as the concentration of ginger essential oil is increased. At a dosage of 110.13 mg/mL, the efficacy of ginger essential oil in scavenging oxidative radicals was found to be 74.19%. The presence of various phenolic compounds (eugenol, zingerone, shogaols, gingerols, *etc.*) plays an important role in the activity against DPPH free radicals [19]. In light of the aforementioned statement, gingerol emerges as a prominent bioactive constituent present in ginger, playing a pivotal role in delivering the characteristic flavour and aroma of this plant. It has been demonstrated that gingerol possesses antioxidant capabilities and is capable of contributing to the neutralization of the free radicals [21].

TABLE-2
OXIDATIVE RADICAL SCAVENGING ABILITY OF GINGER ESSENTIAL OIL ACCORDING TO CONCENTRATION

D (mg/mL)	F	C (mg/mL)	Abs 517	Inhibition (%)
881	50	17.62	0.92667	28.82
881	40	22.03	0.91669	29.59
881	25	35.24	0.81837	37.14
881	20	44.05	0.74640	42.67
881	10	88.10	0.47380	63.61
881	8	110.13	0.33607	74.19

Conclusion

Hydrodistillation is a traditional and effective method for extracting the essential oils from plants. The capacity to capture the inherent fragrance and chemical structure of plant components is highly valued. However, the exact composition of the extracted oil can vary based on factors such as ginger variety, growing conditions and parameters of the extraction process. The factors affecting the distillation process of ginger essential oil on a laboratory scale after optimal conditions include raw materials were dried at 75 ºC for 60 min. Ingredients were pureed in 12 sec and the suitable distillation time was 90 min with the ratio of ingredients:water (w/v) 1:1. The recovery efficiency of ginger essential oil was found to be 2.65 mL/g. The analysis of ginger essential oil revealed that two predominant components were zingiberene, which account for 18.95% of the total content, whereas camphene accounts about 15.265% of the total content. Ginger essential oil has various bioactive components, which are responsible for its possible antioxidant activities. The antioxidant capacity of the ginger essential oil has been determined to be 74.19%.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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