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REGULAR ARTICLE

Microwave assisted Soxhlet extraction of essential oil from Vietnamese Star anise fruits (*Illicium verum* Hook.f.) and their chemical composition

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ABSTRACT

The influence of microwave power (W), irradiation time (h) and particle size (P) on the yield of extraction and composition of essential oil from *Illicium verum* Hook.f has been assessed by means of a new experimental design. The combined effects of three factors above on the yield of extraction of essential oil Y% (r.m) have been determined using Doehlert matrices in the range of 100 - 400 W for microwave power, 10 - 40 mins for irradiation time and 0.4 - 2.0 mm for particle size. In these conditions, second order polynomial relationship between Y% (r.m) and these factors have been established with regression coefficients close to 0.93. The major effect of microwave power and particle size on the yield of extraction of essential oil has been highlighted. In the conditions of 325 W microwave power, 20 min irradiation time and 0.4 mm particle sizes, the highest essential oil yield by microwave assisted Soxhlet extraction was 8.3%. The main compound of *Illicium verum* Hook.f was the (E)-anethole (92 %). The composition of α-pinene, β-mycrene, α-phellandren were absent from the essential oil extracted from *Illicium verum* Hook. f. by use of MASE.

Keywords: Microwave assisted extraction (MAE); Extraction; Essential oil; *Illicium verum* Hook. f.; Star anise fruits; Doehlert matrices

INTRODUCTION

Star anise (*Illicium verum* Hook. f.) fruits is referred to as aromatic evergreen tree. This plant is one of the most important essential-oil trees grown in Northern Vietnam (Wei et al., 2014; Nguyen et al., 2012; Dang and Ilangantileke, 1997; Loi and Thu, 1970. It is often used not only as a flavor in Vietnamese cuisine (Nguyen et al., 2012; Dang and Ilangantileke, 1997; Loi and Thu, 1970) but also an element of the traditional spice powder of Chinese cooking. It is applied in variety of the Vietnam's traditional foods like Pho bo soup (Chempakam and Balaji, 2008). The essential oil extracted from Star anise fruits is applied as aroma in cookies, biscuits and in treating colic and rheumatism (Claus and Tyler, 1965). Star anise has been used as a medicinal plant for a long time in Vietnam. Moreover, the crude fruits and its powder both are used in traditional teas in order to treat nervousness and sleeplessness,etc. The essential oil from this fruits is used for treatment of stomachaches and rheumatism. Recently, the studies have reported that Star anise contents some substances considered antioxidant activities (Kim and Kim, 2003) and significant anti-carcinogenic potential (Yadav and Bhatnagar, 2007). Furthermore, the studies showed that Star anise oil possesses (E)-anethole with concentrating to be about 86.0% - 93.0% (Cook and Howard, 1966; Wichtl, 2004). Besides, it contents other compounds such as estragole, pinene, β-phellandrene, limonene, (Z)-anethole, and α-terpineol (Li and Liu, 2000; Cu et al., 1990).

Normal methods applied to extract Star anise fruits'essential oil have been reported such as hydro-distillation, solvent distillation and steam distillation. The traditional method uses steam distillation. The high temperature and hydrolytic effect of water may cause degradation of essential oil (Yan et al., 2002; Reverchon, 1997). Recently, isolated oils from some plants by supercritical fluid CO_2 and specialty microwave-assisted extraction have been payed attention to researchers and industries.

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Microwaves have frequencies from 300 MHz to 300 GHz, located between the X and infrared rays in the electromagnetic spectrum (Letellier and Budzinski, 1999). Microwave sharply relies on the dielectric susceptibility not merely the solvent but also solid plant matrix (Zuloaga, 1999). Moreover, there were many reports on applicating of microwave in environmental residues (Letellier and Budzinski, 1999; Zuloaga et al., 1999; Sanghi and Kannamkumarath, 2004; Karthikeyan et al., 2006).

Microwave heating plays an important role in analytical and organic laboratory, a very effective and non-polluting activating method. This method is employed in a variety of fields such as digestion, analytical chemistry, organic synthesis, the food industry and so on. Moreover, microwave-assisted extraction is an alternative to normal techniques and the most economical technique to extract compounds from many plants (Hemwinmon et al., 2007). It can reduce not merely the extraction time but also solvent consumption. On the other hand, it has the potential to improve extraction quality (Pan et al., 2008; Pallaronis et al., 2002).

However, there is not any report published about extracting essential oil from Star anise by microwave-assisted extraction. The extraction by microwave technique possesses its own specific parameters which need to be characterized for different essential oil containing plants.

In this paper, the effects of microwave energy, irradiation time and particle size on extraction of Star anise fruits (*Illicium verum* Hook. f.) grown in Vietnam will be determined and extraction parameters will be optimised. A Doehlert's protocol will be used to reduce the number of experiments. Doehlert matrices (Doehlert, 1970) are particular experimental designs which represent the advantage of both the variables space and the experimental space (Quignon et al., 1997). Thus, it is possible applied not simply to appreciate the impact of a first set of experiments with a limited number variables but also to carry out another set of experiments where the impact of the remaining variables is evaluated. Furthermore, the selected range of any parameters tested can be enlarged by presenting another set of experiments, without repeating all former experiments. The combined effects will be presented by means of a polynomial model (Taragano and Pilosof, 1999).

MATERIALS AND METHODS

Collecting sample

Star anise fruits (*Illicium verum* Hook. f.) were collected in Lang Son province, Vietnam from September to October, 2014. The fruits were selected from different trees, about 30 plants per lot. The fruits were cleaned to remove soil and other materials. The fruits were stored in plastic box, kept in ice and transported to the laboratory in the same day. The samples were mixed carefully. The moisture content of Star anise has been modified by low-temperature drying. The final moisture content is 11%. Then the fruits were frozen, freeze-dried and stored at −53°C under nitrogen gas until analysis.

Sample preparation

The Star anise fruits were grinded into 3 particle size fractions of 0.4 mm (mesh no.40), 1.2 mm (mesh no.16) and 2.0 mm (mesh no.10) (American Standard Sieve Series ASTM E11:95).

Microwave assisted Soxhlet extraction (MASE)

The equipment operates similarly to a normal Soxhlet extractor with Model: R106S (i.e. the sample is dissolved in fresh, recycled solvent) the sole difference being that the sample receives microwave irradiation over a present period when it is in contact with the extractant. The multimode microwave reactor has a twin magnetron (2 x 800 W, 2450 MHz) with a maximum delivered power of 1000 W in 10 W increments. A rotating microwave diffuser ensures homogeneous microwave distribution throughout the plasma coated (35 cm x 35 cm x 35 cm). The temperature was controlled by feedback to the microwave power regulator. The sample vessel's temperature was controlled with a microwave-inert optical fire temperature probe and it was not exceed 100°C.

In a typical MASE procedure, 10 g the Star anise fruits was homogenized and placed in a cellulose thimble, which was capped with cotton wool and put into the cartridge vessel located in the zone of microwave irradiation. This device operates with microwave power from 100 W to 400 W. The overall Soxhlet glassware was fitted to a distillation flask, containing 100 ml water and two to three boiling glass regulators. Extraction was operated until no more oil was collected and the solvent was released by using a rotary-evaporator and the oil layer was dried by anhydrous sodium sulfate. The yield was expressed as weight percentage of essential oil of raw material Y (r.m).

Steam extraction

50 g sample was submitted to hydro-distillation with a Clevenger-type apparatus and extracted with 500 ml water in the 1000 ml flask about 120 min. The essential oil was collected to cool at room temperature and the volume was read and stored at 4°C until used. The yield was expressed as weight percentage of raw material (r.m).

Analysis by gas chromatography – mass spectrometry (Fig. 3)

The essential oils were directly analyzed by gas chromatography coupled to mass spectrometry

(Hewlett-Packard 6890 coupled to a Agilent 5973). The column was a HP-5MS (30 m long, 0.25 mm and 0.25 μm film thickness). The operating conditions were as follows: Flow rate 1.0 mL/min; injection temperature 250°C; carrier gas He; split 1:20; injection volume 0.2 μL; oven temperature progress from 60°C to 220°C at 5°C/min, from 220°C to 250°C at 10°C/min; the ionization mode was electronic impact at 70 eV. The constituents in the essential oils were determined by analysing matching of their mass spectral fragmentation patterns with those of compounds in the data bank NIST 98 and Wiley 275 library. Peak normalization method assuming equal detector response has been used to calculate percentage composition. Each sample was duplicated, thus, in total four analyses were performed for extracts for each process. In the percentage compositions with relative standard deviation (R.S.D.) were the average of four analyses.

Experimental design

An experience was defined with microwave power (100 - 400 W), irradiation time (10 - 40 min) and particle sizes (0.4 - 2.0 mm). Each experiment was performed in triplicate. The Doehlert designs are surface response designs, which use to describe **a** region around an optimal response with a polynomial relationship and contains $k^2 + k + 1$ points for k variables. For 3 variables, a set of 13 experiments was performed and one of the properties of the design is the uniform distribution in a three-dimensional space. Each experiment can be located by its three coded values. Thus, 12 experiments are equidistant from a central experiment having the coded values: (0, 0, 0) and are distributed on an area with a radius of 1.

In this study, the yield Y (r.m) was estimated, taking into account the influences of 3 factors (i.e. variables): Microwave power (X_1), irradiation time (X_2), and particle sizes (X_3).

Analysis and interpretation of the results

Multiple regression analysis based on least square method was performed by using the software Nemrod (LPRAI, Marseille, France). The analysis concerned the linear and quadratic effects of the three factors and their interactions. Thus, the equation giving Y was a second-order polynomial model with 10 coefficients (b_0, b_1, b_{12}...b_{23}): $Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3$ where X_1, X_2 and X_3 = factors studied.

The significance of the coefficients was evaluated by multiple regression analysis based upon the F-test with unequal variance, $P < 0.05$ (*), $P < 0.01$ (**) and $P < 0.001$ (***). The response curves which have be drawn for two factors by keeping the third factor at its optimal value obtained through using the same software.

RESULTS AND DISCUSSION

Influence of particle size to yield

The oil yield distilled for four particle sizes was given in Fig. 1. The results showed that particle size sharply effects on the yield. The oil yield for particle sizes about 0.4 mm and from 0.4 to 1.2 mm was not significantly different with both sizes. The highest oil yield was 9.5 (r.m) for particle sizes from 0.4 to 1.2 mm and particle sizes smaller of 0.4 mm. The slowest distillation was a particle size of whole fruit. The results indicated that *Illicium verum* Hook. f. could use the smaller particle size about 1.2 mm.

Effects of microwave power, irradiation time and particle sizes on the yield Y (r.m) by microwave assisted Soxhlet extraction

The Y (r.m) values obtained in the various conditions as reported in this paper are presented in Table 1. The

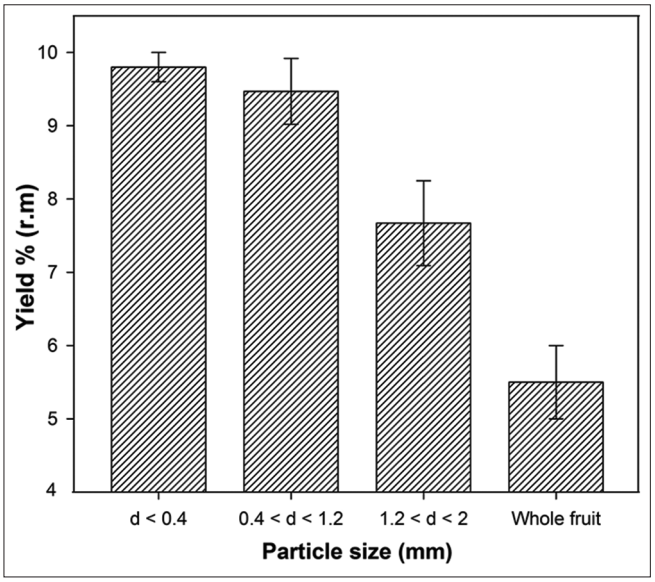

Fig 1. Influence of particle size to yield.

Table 1: Experimental values for Y%

N°Exp	Microwave power, W	Irradiation time, min	Particle sizes, mm	Yield Y % (r.m), mean±sd
1	400	25	1.2	6.17±0.29
2	100	25	1.2	4.50±0.50
3	325	40	1.2	6.17±0.29
4	175	10	1.2	4.17±0.29
5	325	10	1.2	5.27±0.25
6	175	40	1.2	6.27±0.25
7	325	30	2.0	4.17±0.28
8	175	20	0.4	6.00±0.50
9	325	20	0.4	8.30±0.28
10	250	35	0.4	7.00±0.00
11	175	30	2.0	3.30±0.26
12	250	15	2.0	3.37±0.15
13	250	25	1.2	6.30±0.25

experiment number 10 was identical. Moreover, the difference between the assays is 0.1% for yield Y % (r.m) less than equal to 1% for yield. The results of 39 different experiments were ranging from 3.3 to 8.3% (r.m.). The highest essential oil yield was 8.3% with microwave power (325 W), irradiation time (20 min) and particle sizes (0.4 mm) from this extraction. As a result, the smaller the particle size was, the faster the extraction of essential oil yields was.

The results of the regression performing model coefficients were listed in the Table 2. The regression coefficients (r^2) were about 0.932. This means that 93% of the experiments in the matrix could be explained by these models. Moreover, coefficients (b_0) were less different than the experimental values of Y from all the factors such as the factor 0 in experiment 1. Furthermore, the greater the absolute value of the linear coefficients (from b_1 to $b_{3)}$ was the more important the variable influencing the response was. Therefore, the particle sizes sharply impact essential oil yield. On the other hand, the particle sizes play more important than the irradiation time on extracted essential oil yield. Besides, the coefficients, b_1 and b_2 were positive; this means that the response increases along with increasing the variable. In contrast, the coefficients, b_3 was negative. In other words, the response declines with increasing the variable. Moreover, irradiation time and microwave power significantly affected on yield (Y%). However, values ($b_{1,}$ b_2 and b_3) could be ranked which depended on their impact to yield Y% (r.m). To increase yield, the factors must meet the following requirements: Particle sizes (b_3 = -2.14), microwave power (b_1 = 0.94) and irradiation time (b_2 = 0.68).

The first order interaction coefficient, b_{23} was not significant, Table 2. The negative values for b_{11}, b_{22}, b_{33}, b_{12} and b_{13} suggest an antagonistic effect were observed on the process of extraction. The interaction coefficients, b_{11}, b_{22}, b_{33}, b_{12} and b_{13} were significant.

Table 2: Model coefficients obtained

N⁰	Nom	Coefficient	Signif. %
1	b_0	6.27	<0.01***
2	b_1	0.94	<0.01***
3	b_2	0.68	<0.01***
4	b_3	–2.14	<0.01***
5	b_{11}	–0.93	0.0232***
6	b_{22}	–0.76	0.171**
7	b_{33}	–0.94	0.0165***
8	b_{12}	–0.69	0.215**
9	b_{13}	–0.65	0.761**
10	b_{23}	0.44	6.0

***Significant (P<0.01); **significant (P<0.05); *significant (P<0.1); ns: Not significant

Model fitting

By omitting the non-significant coefficients, the reduced equations which described the relationship between the factors and the yield Y % (r.m):

$$Y\ \%\ (r.m) = 6.27 + 0.94\ W + 0.68\ h - 2.14\ P - 0.93\ W^2 - 0.76\ h^2 - 0.94\ P_2 - 0.69\ W.h - 0.65\ W.P$$

In order to determine the optimum conditions for yield Y %, surface response contour plots have been drawn. One factor was fixed arbitrarily at the center of the matrix, while the two other factors were varying. By fixing the particle sizes at 1.2 mm, the effect of microwave power and irradiation time on Y% was shown on Fig. 2. The optimum conditions were schematized as an ellipse. The center of the ellipse (275 - 350 W; 25 - 34 min) represented the optimum conditions in terms of microwave power and irradiation time at particle sizes equal to 1.2 mm. In these optimum conditions, the model predicted yield Y% of essential oil for *Illicium verum* Hook. f. In order to prevent from essential oil extraction, it was therefore recommended to keep away from these optimum conditions. In the experimental matrices schematised by circles were listed in Fig. 2.

Composition of the essential oils

The content and the representative composition of the essential oils of *Illiciumu verum* Hook. f. were pointed in the Table 3. (E)-anethole was the most important compound (92.43%). This result was according to the literature (Cook and Howard, 1966; Wichtl, 2004). The composition of

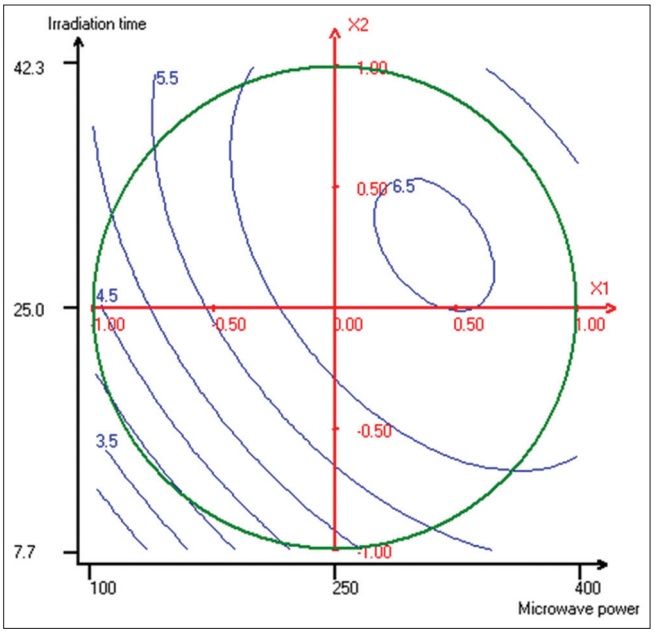

Fig 2. Contour plots of the influence of microwave power and irradation time on Y% (r.m) of essencial oil for *Illicium verum* Hook. f. at particle size=1.2 mm.

Table 3: Retention time and components and content of essential oils extraction from fruit of *Illicium verum* Hook. f. by microwave assisted Soxhlet extraction and steam distillation

N⁰	Compounds*	Retention time, min	Content (%)**	
			Steam distillation	MASE
1	α – Pinene	5.40	0.70±0.2	-
2	β – Myrcene	6.95	0.10±0.1	-
3	α – Phellandrene	7.37	0.24±0.3	-
4	δ - 3 – Carene	7.50	0.46±0.4	-
5	Para – Cymene	7.95	0.20±0.2	-
6	Limonene	8.04	4.34±0.6	-
7	1,8 – cineole	8.16	0.40±0.1	-
8	Linalool	10.33	-	0.33±0.1
9	Terpinen-4-ol	12.90	-	-
10	α-Tecpineol	13.14	0.14±0.1	0.01±0.0
11	4-Allylanisole	13.69	0.38±0.2	0.12±0.1
12	1-Methoxy-4-(1-propenyl)-benzene	15.57	-	0.14±0.1
13	Para-Anisaldehyde	15.63	1.84±0.3	1.65±0.3
14	(E)-Anethole	16.91	89.19±0.7	92.43±0.8
15	α-Copaene	19.50	0.16±0.1	0.15±0.1
16	2-Propanol	19.73	-	0.19±0.1
17	endo-2,6-dimethyl-6-	20.78	-	0.18±0.2
18	β-Caryophyllene	20.87	0.20±0.1	0.19±0.1
19	Trans-α-Bergamotene	21.43	0.55±0.4	0.46±0.3
20	Trans-beta-farnasene	22.13	-	0.13±0.1
21	β-Bisabolene	23.65	0.19±0.1	0.14±0.1
22	1-(3-metyl-2-butenoxy)-4-(1-propenyl)-benzene	28.58	0.77±0.3	0.25±0.2
23	1-butanol	33.03	0.45±0.2	-
24	4-methoxy-benzaldehyde	33.86	0.80±0.3	-

*As identified by GC–MS software; names according to both NIST 98 and Wiley 275 library, **Quantitative data were obtained by relating individual peak areas to the total area of the total ion chromatogram

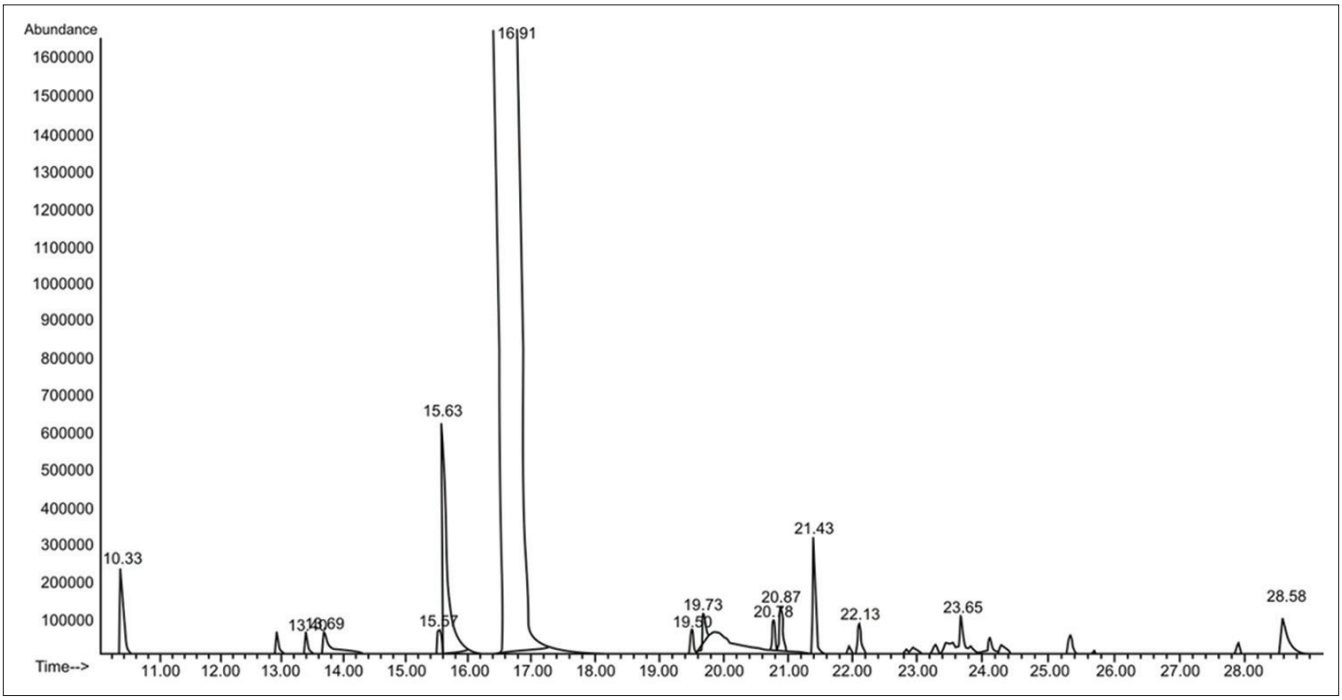

Fig 3. Gas chromatograms of essential oils extraction from fruit of *Illicium verum* Hook. f. by microwave assisted Soxhlet extraction.

the essential oil extracted from *Illicium verum* Hook. f. by MASE was used the same as that obtained by other methods. Although there was some difference in the content fraction of identical compounds, the major

compound, including (E)-anethole were obtained in both methods similarly.

However, the oil's compositions were slightly different in both methods. In fact, if oils are extracted by MASE, some ingredients such as α-pinene, β-mycren, α-phelandren will be absent. Although there were not these substances, this slightly impacted the quality of essential oil, because their content in oil is small.

In this study, the performance of microwave assisted Soxhlet extraction (MASE) technique has been showed. It was reported that particle sizes play an important role for yield oil extracted (Dang and Ilangantileke, 1997). Our results were in accordance with these observations, particle size was more important than other factors to yield.

The yield oil using steam distillation and supercritical fluid CO_2 extraction from *Illicium verum* Hook. f. has achieved 10.2 and 11.2% (r.m), respectively (Dang and Ilangantileke, 1997). In other studies, the essential oil yield by steam distillation was 9.5% (r.m). This could be explained by the differences of particle sizes.

Interestingly, Wang et al. (2006) showed that with a microwave irradiation power of 85 W, it took only approximately 30 min to extract the essential oil completely by solvent-free microwave extraction technique. Recently, Zhai et al. (2009) reported that the extraction time was less than 15 min at the microwave power of 440 W to compared with those obtained by hydro-distillation when using ionic liquid as microwave absorption medium. In the paper, the highest essential oil yield from microwave assisted Soxhlet extraction was 8.3% at 325 W microwave power, 20 min irradiation time and 0.4 mm particle sizes. On the other hand, extraction times was very short compared to other methods such as steam distillation or supercritical fluid CO_2 extraction. Dang and Ilangantileke (1997) reported that it took 1.5 h and 4 h to extract essential oil from Illicium verum Hook. f. by steam distillation or supercritical fluid CO2 extraction process.

Doelhert models were operated to describe optimal conditions for extraction of essential oil from *Illicium verum* Hook. F. These methods were significantly suitable to extract essential oils. Previously, polynomial models were used to describe extracted essential oil from *Elettaria cardamomum* (L) (Lucchesi et al., 2007). The multifactorial analysis was essential to develop a model, an appropriate model to describe possible interaction between the factors

The quality of essential oil obtained by MASE technique was similar to using steam distillation. Moreover, the MASE technique recovered less volatile constituents than conventional distillation but these constituents did not significantly contribute to final profile of flavour such as: α - pinene, β - mycren, limonene.

CONCLUSION

In this work, essential oil was extracted by the microwave assisted Soxhlet extraction methods. The optimal conditions included in the following parameters: Microwave power 325 W, irradiation time 20 min and particle sizes 0.4 mm. Maximum essential oil yield was 8.3%. The main compound of *Illicium verum* Hook.f was the (E)-anethole (92 %). Moreover, the results obtained in the study, will provide basic data for further development and application of star anise for food industry. In tropical countries, with variety of vegetation resources, this method was very capable of developing.

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Author's contributions

N. N. Phan, L. T. K. Phan and T. H. Tran participated in the experiments and data analysis, T. Dao, the corresponding author designed the research plan, organized the study, and contributed to the writing of the manuscript. M. H Chatain participated in the experimental design and also contributed to the writing of the manuscript.

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