

Journal of Tropical Pharmacy and Chemistry

Journal homepage: https://jtpc.farmasi.unmul.ac.id

# The Optimization of Essential Oil Extraction from Java Cardamom

Raissa, Windi Cahya Amalia, Meri Ayurini, Khabib Khumaini, Paramita Jaya Ratri\*

Universitas Pertamina, Indonesia \*E-mail: <u>paramita.jr@universitaspertamina.ac.id</u>

# Abstract

Indonesia is one of the largest spice-producing regions such as the Java Cardamom (Amonum *compactum*). The cardamom can be converted into derivative products in the form of cardamom essential oil. In this work, we attempted to extract the essential oil from the cardamom by comparing two methods of extraction which are the steam distillation and the simple distillation. The optimization factors considered on the extraction yield were solvent (ethyl acetate and n-hexane) and extraction time (3-6 h). The extraction yield obtained by both methods in ethyl acetate solvents was almost equal to that obtained in n-hexane. Also, the result obtained revealed that the extraction yield increased with time. The optimum essential oil yield was obtained by the simple distillation method in 6 hours duration time using n-hexane solvent giving the yield of 6.3 %. Gas Chromatography-Mass Spectrometry (GC-MS) was used to identify the chemical composition of the extracted oil. The results showed that the chemical composition of the essential oils is different in each extraction time. The main compounds in all oil samples were eucalyptol and camphene among four other constituents. The concentration of eucalyptol reached an optimum (90.89-93.74 %) at 4-5 h of distillation times, while the concentration of camphene reached an optimum (52.98 %) at 6 h. The purity of the essential oil was confirmed by Fourier Transform Infra-Red (FTIR). Moreover, this research will help to utilize the cardamom due to its main compounds that act as herbal medicine.

Keywords: Cardamom, Amomum compactum, Essential Oil, Extraction, Steam Distillation, Simple Distillation

Submitted: 24 April 2020

Accepted: 06 August 2020

DOI: https://doi.org/10.25026/jtpc.v5i2.257

## Introduction

For centuries, Indonesia has been prominent as one of the world's spice producers. Clove, nutmeg, mace, cinnamon, and cardamom are a variety of spices grown in Indonesia. Amomum compactum or Java Cardamom is one of the main cardamom species dominated in Indonesia [1]. It is well-known for flavor, medicinal, fragrant, anti-inflammatory [2], anti-microbial [3], and antioxidant attributed to the presence of essential The Optimization of Essential Oil Extraction from Java Cardamom

oils. The extraction of essential oil from Java Cardamom has been reported by hydrodistillation [4], microwave-assisted hydrodistillation [4], and steam distillation methods. Some reports found that eucalyptol or 1,8 cineole [1], [5] is the main compound of the cardamom, while  $\alpha$ -pinene,  $\alpha$ terpineol, and humulene are also obtained in smaller doses. However, to the best of our knowledge, no studies have been reported in the literature about a comparative study of the oil composition from the Amomum compactum in different distillation times. Therefore, in this work, we extracted the essential oil by steam distillation technique at various times. Moreover, the work aims to find the effect of two methods which is steam distillation and simple distillation methods on the yield of essential oils.

## Experimental

The cardamom used in this research is Java cardamom or Amomum compactum purchased from the local market of Jakarta, Indonesia. The cardamoms were dried, peeled, and grounded into a coarse powder. In distillation methods, n-hexane (Merck,  $\geq$  96%) and ethyl acetate (Merck,  $\geq$  99.5%) were used as solvents.

The distillation experiment using 20 g of the cardamom powder was conducted at Universitas Pertamina in 2019. The essential oil was extracted by two methods which were the steam distillation and the simple distillation methods as described in previous reports [6], [7], [8]. The steam distillation and the simple distillation was carried out in 250 mL-steam distillation unit with n-hexane and ethyl acetate as solvents. The distillation time for the steam distillation technique tested in this research was 3, 4, 5, and 6 hours, while the extraction time via the simple distillation was 6 h. All distillation times were measured from the first drop of essential oil until the heating was turned off.

The essential oils of the cardamom were done by gas chromatography-mass spectrometry (GC-MS). The system had a gas chromatograph and equipped by a mass spectrometer (TraceGoldTM 1310) with TG-1MS fused silica capillary tubes columns (length 30 m; ID 0.25 mm; film thickness 0.25 m). The spectra were obtained by the following conditions: carrier gas, He with a flow rate of 1 ml/min; the split ratio was 1:40; injection volume was 1  $\mu$ L, injection temperature was 250 °C; oven temperature was 50 °C, it increased at a rate of 10 °C/min to 280 °C and held at 230 °C for 1 min then 280 °C for 10 min; ionization mode, electron energy was at 70 eV, emission current was 50  $\mu$ A.

Fourier transform infra-red spectroscopy (FTIR) analysis was performed using Thermo Scientific, Nicolet IS5 with iD5 ATR mode. Essential oils were piped into the sample holder. The spectra were obtained in the spectral region 4000 to  $500 \text{ cm}^{-1}$  with the resolution  $4 \text{ cm}^{-1}$ .

# Results and Discussion

The work was carried out by comparing two different methods which are the steam distillation and the simple distillation methods at different parameters condition to obtain a maximum yield of essential oil.

### Effect of solvent on yield

Effect of solvent on the extraction yield of Java Cardamom at 200-250 °C for 6 hours shown in Table 1. From the graph, it can be concluded that ethyl acetate and n-hexane solvents gave similar yield in the steam distillation and the simple distillation methods. Also, it was observed that from Table 1, the extraction yield of the oil by the simple distillation method was found to be greater than the steam distillation method.

Table 1. Extraction yield of essential oil comparing using three extraction techniques in two different solvents at 200-250 °C for 6 hours

Extraction Method	Extraction Yield (% w/w)		
Extraction Method	Ethyl Acetate	n-hexane	
Simple Distillation	5.9	6.3	
Steam Distillation	5.6	5.6	

## Effect of extraction time on yield

The extraction time was varied to examine the effect of time on the yield of essential oil from Java Cardamom. Figure 1 shows the yield of the extracted oil at different extraction times using the steam distillation method in n-hexane at 300-315 °C. The steam distillation was performed rather than the simple distillation due to its ability to purify the temperature sensitive compound inside the cardamom. It was found that the essential oil yield was low at 3-4 h of distillation times, increased at 5 h, and reached the highest yield at 6 h. In this case, the extraction yield increased with time, however, further research is needed to examine the effect of distillation time above 6 h.

#### Analysis of GC-MS and FTIR spectrum

Essential oils from the Java Cardamom with the steam distillation method are analyzed by GC-MS to analyze the constituents which are present in the oil in different extraction times. The five components identified from the essential oil of Java Cardamom are eucalyptol or 1,8 Cineole, camphene, terpinen-4-ol, a-terpinol, terpynil acetate, and eugenol. The constituents and their percentages are summarized in Table 2.

The analysis of the results shows that the chemical composition of the essential oils is different in each extraction time. The essential oil obtained at 3 hours of extraction time gives eucalyptol (33.65 %) and camphene (23.40 %) as the major constituents. The later fractions are rich in Eucalyptol giving the percentage of 90.89 % and

93.74 % at 4 and 5 hours of extraction times respectively. At 6 hours, camphene (52.98 %) is detected as the primary component. Overall, the result shows that eucalyptol and camphene are the key compounds in all samples.

Our findings were quite different with the report by Xue Feng et al showing that camphene (0.2 %) was a minor constituent in the essential oil of the Amomum compactum extracted by microwave-assisted hydrodistillation technique [4]. In this work, high percentages of camphene (Table 2) were obtained by the steam distillation method at 3 h and 6 h of extraction times. Camphene is a monoterpene whose empirical formula is  $C_{10}H_{16}$  [9] that can be anti-oxidant [10], anti-fungi [11], pain reliever [12], and lipidlowering agent [13]. It is present in numerous plant such as sage and lavender. The presence of eucalyptol and camphene was consistent with the FTIR spectrum (Figure 2) as the peaks at 1100 – 1200 cm<sup>-1</sup> and 2950 cm<sup>-1</sup> corresponding to the group of eucalyptol and aliphatic ether hydrocarbons camphene respectively.

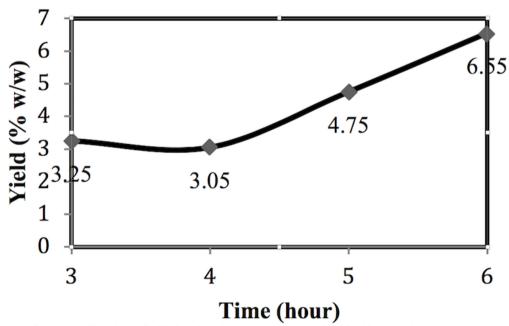


Figure 1. Graphic of essential oil yield vs distillation time that shows the extraction yield increased with time.

The Optimization of Essential Oil Extraction from Java Cardamom

Retention Time (min)	Compounds	Area percent (%)			
		3 hours	4 hours	5 hours	6 hours
7.34	Eucalyptol	33.65	90.89	93.74	11.52
11.97	Camphene	23.40	5.35	3.32	52.98
9.57	Terpinen-4-ol	9.14	1.41	1.24	7.51
9.57	a-terpinol	6.75	1.40	1.22	14.49
11.53	Terpynil Acetate	11.43	0.58	0.40	5.16
12.07	Eugenol	15.63	0.37	0.08	8.33
	Total	100	100	100	99.99

Table 2. The essential oil composition of the Java Cardamom essential oils of Java Cardamom in different extraction times via GC-MS analysis

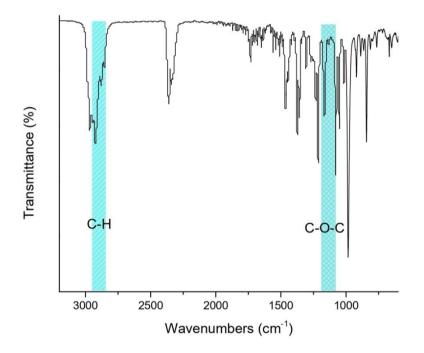


Figure 2. FTIR Spectrum of the cardamom essential oil that confirms the presence of eucalyptol and camphene as the peaks at  $1100-1200 \text{ cm}^{-1}$  and 2950 cm<sup>-1</sup> consistent with the ether group of eucalyptol and aliphatic hydrocarbons camphene respectively

### Conclusion

In present work, essential oils of the *Amomum compactum* were successfully extracted by steam and simple distillation methods at various extraction times. Among all these experiments, steam distillation at 6 h of extraction time using nhexane solvent gave the highest percentage yield. Extraction time has been shown to effect on essential yield and composition. Moreover, evidence from GC-MS and FTIR analysis showed that eucalyptol and camphene were the main compounds in the oil.

#### References

- M. Nesbitt, C. C. de Guzman, and J. S. Siemonsma, 2007. Plant Resources of South East Asia No. 13. Spices, *Kew Bulletin*.
- [2] J. A. Lee, M. Y. Lee, I. S. Shin, C. S. Seo, H. Ha, and H. K. Shin, 2012. Anti-Inflammatory Effects of Amomum Compactum on RAW 264.7 Cells via Induction of Heme Oxygenase-1, *Archives of Pharmacal Research*, **35**, (4), 739–746.
- [3] M. Simsek and R. Duman, 2017. Investigation of Effect of 1,8-Cineole on Antimicrobial Activity of Chlorhexidine Gluconate, *Pharmacognosy Research*, 9, (3), 234–237.
- [4] Z. T. Jiang, X. Feng, R. Li, and Y. Wang, 2013. Composition Comparison of Essential Oils

The Optimization of Essential Oil Extraction from Java Cardamom

Extracted by Classical Hydro Distillation and Microwave-Assisted Hydrodistillation from Pimenta Dioica, *Journal of Essential Oil-Bearing Plants*.

- [5] K. H. Timotius, I. N. Sari, and A. W. Santoso, 2015. Major Bioactive Compounds of Pilis Plant Materials: A GC-MS Analysis, *Pharmacognosy Communications*, 5, (3), 190–196.
- [6] M. T. Golmakani and K. Rezaei, 2008. Comparison of Microwave-Assisted Hydrodistillation Withthe Traditional Hydrodistillation Method in the Extractionof Essential Oils from Thymus Vulgaris L., *Food Chemistry*, **109**, (4), 925–930.
- [7] L. L. Dilworth, C. K. Riley, and D. K. Stennett, Plant Constituents: Carbohydrates, Oils, Resins, Balsams, and Plant Hormones, in *Pharmacognosy: Fundamentals, Applications and Strategy*, Jamaica: Elsevier Inc., 61–80.
- [8] M. Golmohammadi, A. Borghei, A. Zenouzi, N. Ashrafi, and M. J. Taherzadeh, 2018. Optimization of Essential Oil Extraction from Orange Peels Using Steam Explosion, *Heliyon*, 4, (11), 1–18.
- [9] T. J. Zachariah and N. K. Leela, Volatiles from Herbs and Spices, in *Handbook of Herbs and Spices*, Woodhead Publishing, 177–218.

- [10] M. Tiwari and P. Kakkar, 2009. Plant Derived Antioxidants - Geraniol and Camphene Protect Rat Alveolar Macrophages against t-BHP Induced Oxidative Stress, *Toxicology in Vitro*, 23, (2), 295–310.
- [11] G. I. K. Marei, M. A. Abdel Rasoul, and S. A. M. Abdelgaleil, 2012. Comparative Antifungal Activities and Biochemical Effects of Monoterpenes on Plant Pathogenic Fungi, *Pesticide Biochemistry and Physiology*, **103** 56– 61.
- [12] J. D. Adams, The Use of California Sagebrush (Artemisia Californica) Liniment to Control Pain, *Pharmaceuticals*. Pharmaceuticals, 1045–1053, 2012.
- [13] I. Vallianou, N. Peroulis, P. Pantazis, and M. Hadzopoulou-Cladaras, 2011. Camphene, a Plant-Derived Monoterpene, Reduces Plasma Cholesterol and Triglycerides in Hyperlipidemic Rats Independently of HMG-CoA Reductase Activity, *PLoS ONE*, 6, (11).