

EFFECTS OF DILUTION AND AGITATION ON EUCALYPTUS PELLITA ESSENTIAL OIL EXTRACTION

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Abstract

Eucalyptus pellita essential oil is commonly extracted by hydrodistillation; however, this process has limited efficiency due to heat and mass-transfer constraints. Process parameters, such as solvent loading and agitation, offer promising but understudied opportunities for process intensification to improve oil yield and quality. This study aimed to analyze the interactive effects of the leaf-to-solvent mass ratio and agitation speed on the extraction yield and chemical profile of E. pellita essential oil obtained through hydrodistillation. Extraction was performed using 150 g of chopped E. pellita leaves with particle sizes of 0.1–0.5 cm at 100°C for 4 hours under atmospheric pressure. The independent variables were leaf-to-solvent mass ratios of 1:4, 1:6, and 1:8, and agitation speeds of 300, 600, and 900 rpm. The extracted oil was quantified based on yield and chemical composition. The chemical profile of the essential oil was characterized using gas chromatography–mass spectrometry (GC–MS). The results showed that solvent proportion had a more significant effect than agitation speed. The 1:8 ratio produced the highest oil mass of 0.62 g and yield of 0.41%. GC–MS analysis identified α -pinene, eucalyptol (1,8-cineole), L- α -terpineol, caryophyllene, and bicyclo[3.1.1]heptane as the major components. This study demonstrates the decoupled effects of solvent ratio and agitation in E. pellita hydrodistillation, revealing the solvent ratio as a chemical driver of compositional selectivity and agitation speed as a physical driver of system homogeneity. This distinction provides a targeted strategy for simultaneously optimizing extraction yield and oil quality.

Keywords: agitation speed; essential oil; *Eucalyptus pellita*; hydrodistillation; solid–liquid ratio.

Introduction

Essential oils are volatile and aromatic compounds produced by various plant parts, such as leaves, flowers, stems, and roots. These oils have been widely used in the pharmaceutical, cosmetic, food, and agricultural industries because of their antibacterial, antifungal, and therapeutic properties [1]. Among the various essential oil-producing plants, *Eucalyptus pellita* is a fast-growing species known for its high

content of bioactive compounds, particularly 1,8-cineole and α -pinene, which contribute to its characteristic aroma and biological activity [2]. However, the potential use of *E. pellita* leaves, which are often discarded as waste by the pulp and paper industry, remains underutilized. Utilizing these residues as raw materials for essential oil extraction can enhance

resource efficiency and promote sustainable production.

Essential oil extraction can be carried out using several methods, such as steam distillation, solvent extraction, and hydrodistillation. Hydrodistillation is widely applied because of its simplicity, efficiency, and ability to produce oil with good purity without significant chemical degradation [3]. Previous studies have shown that process variables, such as the solid-to-liquid ratio, agitation speed, and extraction time, strongly influence the efficiency of essential oil recovery [4], [5]. Lainez-Cerón et al. [6] reported that variations in agitation speed and solvent ratio can alter the yield and composition of eucalyptus essential oil by affecting the rate of mass transfer and the stability of the emulsion formed during distillation.

In the hydrodistillation process, the leaf-to-solvent mass ratio determines the contact surface area between the plant material and the liquid phase, whereas agitation speed ensures temperature and mass-transfer homogeneity within the system. An optimal balance between these two parameters is essential to obtain high yields and preserve the integrity of volatile components. Therefore, this study aimed to evaluate the effect of the leaf-to-solvent ratio and agitation speed on the yield and chemical composition of essential oil extracted from *E. pellita* leaves using the hydrodistillation method.

Experimental section

Materials

Fresh *E. pellita* leaves aged approximately five years were obtained from PT Tanjungenim Lestari Pulp and Paper, South Sumatra, Indonesia. The leaves were used as the raw material for essential oil extraction, as shown in Figure 1. Distilled water was used as the solvent in all hydrodistillation experiments.



Figure 1. *E. pellita* leaves.

Instruments

The hydrodistillation apparatus used in this study is shown in Figure 2. The system consisted of a magnetic hotplate stirrer (1), a flat-bottom distillation flask (2), a Clevenger-type apparatus (3), a condenser (4), cooling water hoses (5), a thermometer (6), and a water reservoir (7) used to supply cooling water during the distillation process.

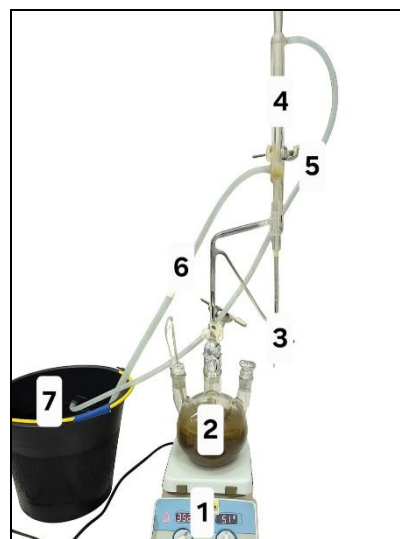


Figure 2. Hydrodistillation apparatus setup.

The hydrodistillation process was conducted using a Clevenger-type apparatus consisting of a 2000 mL flat-bottom flask, a condenser, and a graduated receiver tube. The system was mounted on a magnetic hotplate stirrer to maintain stable heating and agitation during extraction. Additional instruments included a stainless-steel grinder, thermometer, analytical balance

(± 0.001 g), and dark glass vials (5 mL) for storage.

Chemical composition analysis of the essential oil was performed using gas chromatography–mass spectrometry (GC–MS) at the Laboratory of Analytical Chemistry and Instrumentation Testing, Faculty of Mathematics and Science, Universitas Sriwijaya.

Procedure

Leaf Preparation

The leaves were washed with clean water, separated from the central veins, and chopped into small pieces measuring 0.1–0.5 cm. A total of 150 g of prepared leaves was used for each experimental run.

Hydrodistillation Process

The variations in leaf-to-solvent mass ratio applied in this study are summarized in Table 1. The ratios were calculated based on a fixed mass of 150 g of prepared leaves for each experimental run.

Table 1. Leaf-to-solvent mass ratios used in the hydrodistillation process

Leaf-to-solvent ratio	Mass of leaves (g)	Mass of distilled water (g)
1:4	150	600
1:6	150	900
1:8	150	1200

The chopped leaves (150 g) were placed into the distillation flask and mixed with preheated solvent according to the leaf-to-solvent mass ratios listed in Table 1. The mixture was heated to boiling and maintained under atmospheric pressure for 4 hours at an operating temperature of 100°C. Agitation speeds of 300, 600, and 900 rpm were applied during extraction to evaluate their effect on oil recovery. Cooling water at 4°C was circulated through the condenser to ensure efficient vapor condensation. The distillate collected in the Clevenger apparatus separated into hydrosol and essential oil layers. The oil layer was collected using a micropipette and stored in amber vials at 2–4°C to prevent

volatilization. A schematic diagram of the overall hydrodistillation procedure is presented in Figure 3 to provide a clear overview of the experimental sequence.

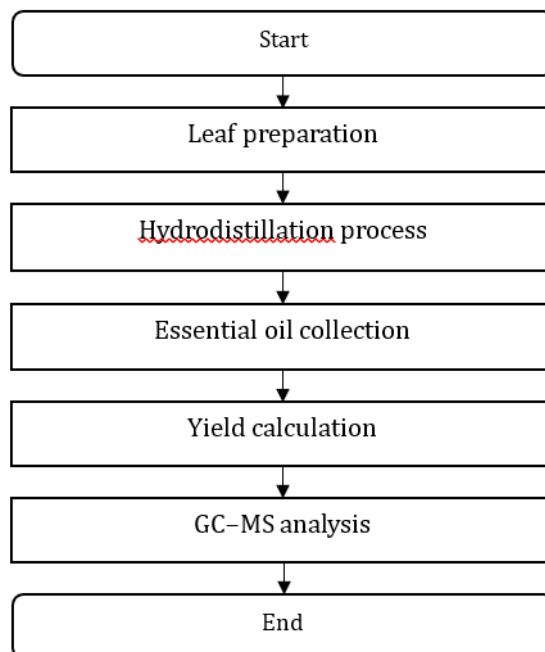


Figure 3. Schematic diagram of the procedure.

GC–MS Analysis

GC–MS analysis was conducted using a Thermo Scientific TSQ 9000. The injector temperature was set at 200°C in splitless mode. The oven temperature was programmed from 60°C for 1 min to 280°C at 5°C/min with intermediate holds. Helium was used as the carrier gas at a constant flow rate of 1.2 mL/min. The MS transfer line and ion source temperatures were set at 290°C and 280°C, respectively. The MS was operated in electron ionization (EI) mode with a scan range of 50–600 m/z. Two representative samples corresponding to the minimum and maximum values of the studied variables were selected for GC–MS analysis. Compound identification was performed using mass spectral library matching (mainlib) with a similarity index (SI) $\geq 90\%$.

Calculation of Yield

Essential oil yield was calculated based on the ratio of the obtained oil mass to the initial mass of leaves used, following

the method reported by Katekar et al. (2023), as expressed in Equation (1).

$$\text{Yield (\%)} = \frac{\text{Mass of extracted oil (g)}}{\text{Mass of leaves (g)}} \times 100 \quad (1)$$

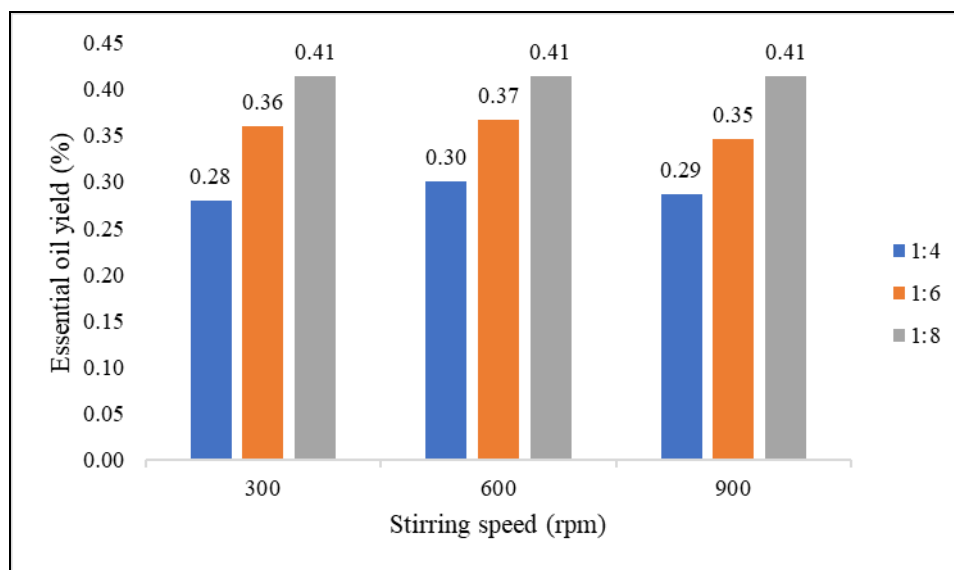


Figure 4. Yield of essential oil at various leaf-to-solvent mass ratios and agitation speeds.

Results and Discussion

Essential Oil Yield

The yield of essential oil exhibited a clear increasing trend with higher leaf-to-solvent mass ratios, as shown in Figure 4. The 1:8 ratio consistently produced the highest yield of 0.41% at all agitation speeds, indicating that solvent volume was the most influential parameter governing hydrodistillation performance. A greater solvent volume enhances mass transfer by increasing the wetted surface area of plant tissues, improving water penetration into oil glands, and promoting thermal softening of cell walls, thereby facilitating the release of volatile compounds [4]. This phenomenon is supported [6], who reported that higher solvent ratios reduce internal resistance during vapor-liquid transfer and promote more efficient transport of essential oil components into the vapor phase.

The consistent yield at the 1:8 ratio also demonstrates the stabilizing effect of solvent volume on system temperature. Larger solvent volumes function as thermal buffers, maintaining a near-constant boiling profile and preventing localized overheating

that could degrade thermolabile constituents, such as eucalyptol and α -pinene [5]. This stabilizing effect ensures that volatile compounds are gradually released and carried by the generated steam without undergoing structural breakdown, resulting in higher oil recovery.

Conversely, variations in agitation speed from 300 to 900 rpm produced only marginal changes in yield. The small differences observed suggest that agitation contributes primarily to temperature uniformity and the prevention of settling rather than directly increasing the release of essential oil. Once the hydrodistillation system reaches a steady-state temperature, additional agitation provides minimal benefit to vapor formation or volatile transport, which is consistent with the findings of [3]. In fact, excessive agitation may generate stable emulsions in the distillate, entrapping oil droplets within the aqueous phase and inhibiting separation in the Clevenger apparatus, a mechanism also described by [7].

Terpene Distribution

The terpene distribution of samples A and B further supports the influence of

hydrodistillation conditions on extraction characteristics, as shown in Table 2. Sample A, obtained using a 1:4 leaf-to-solvent ratio and an agitation speed of 300 rpm, contained 47.52% oxygenated monoterpenes and 29.33% hydrocarbon

monoterpenes, confirming that lighter and more volatile constituents dominated under lower solvent conditions. This finding is consistent with the rapid co-distillation behavior of monoterpenes, which typically evaporate early during hydrodistillation [8].

Table 2. Terpene distribution of *E. pellita* essential oil from samples A and B

Terpene class	Sample A (% area)	Sample B (% area)
Monoterpenes	29.33	27.09
Oxygenated monoterpenes	47.52	39.46
Sesquiterpenes	14.09	22.17
Oxygenated sesquiterpenes	8.44	10.59
Esters/other compounds	0.65	0.68
Total (% area)	100	100

In contrast, Sample B, obtained using a 1:8 leaf-to-solvent ratio and an agitation speed of 900 rpm, exhibited a more chemically diverse profile, with higher proportions of sesquiterpenes (22.17%) and oxygenated sesquiterpenes (10.59%). Sesquiterpenes generally have higher molecular weights and boiling points, requiring prolonged exposure to hot solvent for effective extraction. Their enrichment in Sample B suggests that the larger solvent volume improved penetration into plant tissues, allowing deeper-seated glandular structures to release heavier compounds, a mechanism supported by [2] and [9].

However, variations in solvent ratio and stirring speed did not produce a significant difference in the total number of identified compounds, with 50 compounds detected in Sample A and 49 in Sample B. This indicates that the hydrodistillation conditions primarily affected relative abundance rather than the presence or absence of individual components. The overall essential oil (EO) chemical profile

remained qualitatively similar between samples, suggesting that the core composition of the oil was preserved across the studied conditions. Despite the shift in terpene class distribution, the number of detected constituents remained comparable between samples, reinforcing that process conditions influenced compositional ratios rather than expanding chemical diversity.

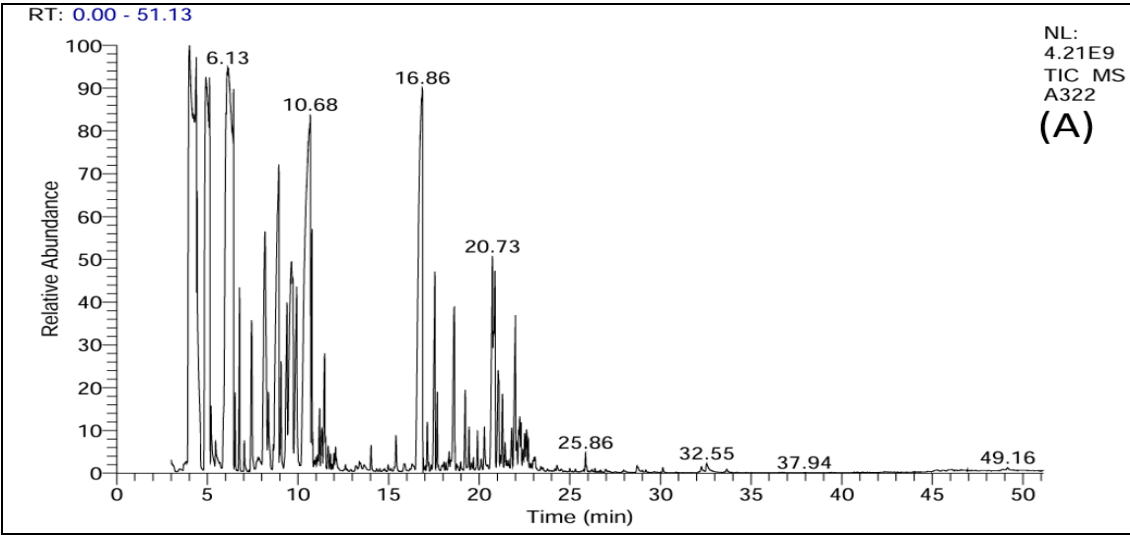
The decrease in the oxygenated monoterpene-to-oxygenated sesquiterpene ratio from Sample A (5.63:1) to Sample B (3.73:1) reveals a shift toward the extraction of more thermally stable oxygenated components. This compositional change aligns with the higher overall yield obtained at the 1:8 ratio, indicating that solvent availability modulates not only extraction efficiency but also the balance of terpene classes [6]. Thus, the effect of hydrodistillation conditions is better reflected in the monoterpene-to-sesquiterpene ratio than in changes to the qualitative EO profile.

Overall, A represents an oil richer in light Overall, Sample A represents an oil richer in light monoterpenes, whereas Sample B captures a wider spectrum of heavier and oxygenated compounds. These differences suggest that solvent volume is a key determinant of terpene group distribution, influencing volatility, solubility, and diffusional transport during

hydrodistillation [5], [10]. The absence of significant variation in the number of identified components confirms that hydrodistillation parameters predominantly govern the proportional distribution of terpene classes rather than altering the fundamental chemical identity of the essential oil.

Table 3. Major chemical constituents of *E. pellita* essential oil from samples A and B

Component	Sample A (%)	Sample B (%)
α -Pinene	17.56	16.7
Eucalyptol	15.69	19.74
L- α -Terpineol	11.09	7.33
Bicyclo[3.1.1]heptane, 6,6-dimethyl-2-methylene-, (1S)	9.22	6.58
Caryophyllene	9.1	1.52



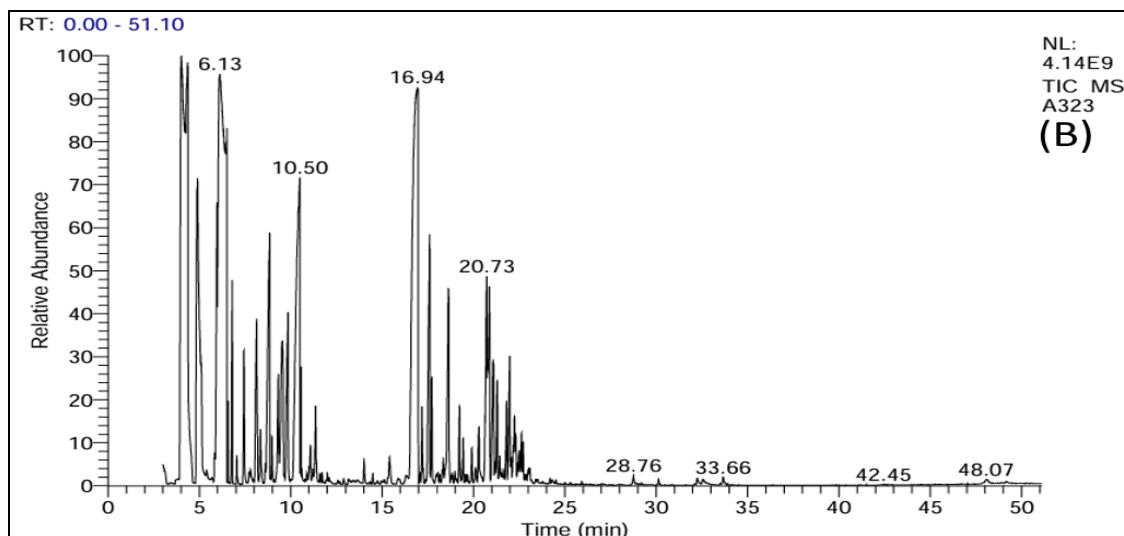


Figure 5. GC–MS chromatograms of samples A and B.

Chemical Composition

Samples A and B were prepared under different hydrodistillation conditions to evaluate the influence of operating parameters on the chemical behavior of the extracted essential oil. Sample A was obtained at a lower leaf-to-solvent mass ratio (1:4) with mild agitation (300 rpm), whereas Sample B was produced under higher solvent loading (1:8) and stronger agitation (900 rpm). GC–MS analysis showed that both samples were characterized by the same major constituents, namely α -pinene, eucalyptol, L- α -terpineol, caryophyllene, and bicyclo[3.1.1]heptane derivatives, as shown in Table 3. These results are consistent with the dominant components of eucalyptus essential oils reported by [9], [11]. This indicates that variations in the leaf-to-solvent ratio and agitation speed did not significantly affect the qualitative chemical profile of the oil.

Although the identities of the major compounds remained unchanged, differences were observed in their relative peak areas, as shown in the GC–MS chromatograms in Figure 5. These differences reflect shifts in proportional abundance rather than the formation or disappearance of specific constituents. The variations are primarily attributed to differences in extraction kinetics, vapor-liquid equilibrium, and mass-transfer

behavior during hydrodistillation [4], [8]. Under lower solvent availability, as applied in Sample A, lighter and more volatile monoterpenes tend to be released and distilled preferentially during the early stages of the process. In contrast, the higher solvent volume used in Sample B enhances tissue swelling and solvent penetration into glandular structures, facilitating the gradual release of less volatile and higher-molecular-weight compounds [12].

The observed changes in relative peak areas, as shown in the GC–MS chromatograms in Figure 5, can also be explained by variations in solubility and diffusional transport under different hydrodistillation conditions. A larger solvent volume promotes more uniform heat distribution and prolonged contact between plant material and hot water, allowing compounds located deeper within plant tissues to diffuse more effectively into the vapor phase [6], [10]. Consequently, heavier and more thermally stable components contribute more substantially to the overall essential oil composition without altering the number or identity of detected compounds.

Overall, these findings demonstrate that hydrodistillation parameters primarily regulate the proportional distribution of major terpene classes rather than altering the fundamental chemical identity of the essential oil. The consistency of major components across samples confirms the

robustness of the eucalyptus essential oil profile, with solvent volume acting as the dominant factor and agitation speed playing a secondary role in controlling extraction dynamics [3], [8].

Conclusion

The results of this study demonstrate that the leaf-to-solvent mass ratio is the most influential parameter governing the hydrodistillation of *E. pellita* essential oil compared with agitation speed. The highest yield of 0.41% was achieved at the 1:8 ratio, indicating that increasing solvent volume enhances mass transfer, stabilizes boiling conditions, and facilitates the release of both light and heavy volatile compounds. In contrast, agitation speed showed a minimal effect on yield, suggesting that its role is limited to improving thermal homogeneity rather than accelerating extraction.

Chemical analysis revealed that both samples were dominated by α -pinene, eucalyptol, L- α -terpineol, caryophyllene, and bicyclo[3.1.1]heptane derivatives, with compositional shifts influenced by solvent availability. The 1:8 ratio promoted the extraction of oxygenated monoterpenes and sesquiterpenes, resulting in a more chemically diverse oil. Overall, the findings confirm that solvent loading not only improves extraction efficiency but also modulates terpene distribution, offering important insights for optimizing the hydrodistillation of eucalyptus-based essential oils.

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