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The Effect of Pretreatment Type on the Yield of Essential Oils from *Eucalyptus* Species (*Camaldulensis And Torelliana*) Using Steam Distillation

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ABSTRACT: In recent years, the challenge of low yield or inferior physicochemical and phytochemical characteristics of oil during the extraction of essential oils from diverse medicinal plants utilizing different methodologies and pretreatment procedures has been the pivot of many studies. In line with this, the study aims to examine how different pretreatment types affect the production and quality of essential oils extracted from *Eucalyptus camaldulensis* and *Eucalyptus torelliana* using steam distillation. The leaves of the two readily available eucalyptus species in Nigeria were first pretreated by chilling at -4°C, airdrying or direct usage as wet leaves and were extracted by indirect steam distillation at a temperature of 100°C and different times of 30-182 min for *Eucalyptus camaldulensis* and 30-240 min for *Eucalyptus torelliana*. The results showed that the highest yield of oil was observed for the chilled pretreated leaves for the *Eucalyptus camaldulensis* (1.4% at 135 min) and *Eucalyptus torelliana* (1.73%, 224.25 min) with *Eucalyptus torelliana* showing a higher yield at a longer extraction time. Consequently, the chilled pretreatment enhanced oil release by disrupting the plant cell structure via freezing. This study indicates that pretreatment methods significantly influence the yield and chemical composition of *Eucalyptus* essential oil derived from *Eucalyptus camaldulensis* and *Eucalyptus torelliana*.

KEYWORDS: *Eucalyptus camaldulensis*, *Eucalyptus torelliana*, essential oils, pretreatment, steam distillation.

1.INTRODUCTION

The genus *Eucalyptus*, part of the Myrtaceae family, includes more than 500 species. It is found in numerous tropical regions, including Nigeria. *Eucalyptus* trees, originally native to Australia, have been spread globally due to their quick growth, adaptability, and versatile applications [1]. Out of the versatility of these plants, the essential oils obtained stand out and it is largely constituted of a mixture of hydrocarbons and oxygenated chemicals such as terpenes, alcohols, esters, aldehydes, and ketones which contribute not only to its smell but also to its biological activity [2].

Eucalyptus oil is notable for its medicinal characteristics, which encompass antiviral, antibacterial, antifungal, anti-inflammatory, and decongestant actions [3]. The bioactivities of Eucalyptus oil render it a significant natural therapy for respiratory disorders, muscular pain, and specific dermatological issues [4]. However, these oils are underutilized, especially in developing countries like Nigeria where a majority of the Eucalyptus oil utilized within the pharmaceutical, cosmetic, and food sectors is mostly imported. This challenge necessitates for study focused on developing locally adapted and effective extraction methods to diminish import reliance and enhance industrial sustainability.

One of the challenges hindering local production of *Eucalyptus* oil is the extraction process which is expensive and sensitive to process conditions and has a vital role in defining the chemical composition, yield, and quality of the oil [5]. In recent years, multiple studies [6] have concentrated on extracting essential oils from diverse medicinal plants utilizing different methodologies and pretreatment procedures. However, a considerable problem persists in generating large oil outputs while retaining the oil's beneficial components. Many of the current procedures applied for Eucalyptus oil extraction have resulted in either low yield or essential oils with inferior physiochemical and phytochemical characteristics. The variations in yield are often linked to the type of pretreatment done to the plant material before extraction [7].

Steam distillation, in particular, is the most extensively used process for extracting the essential oils. This approach is recommended because it permits the volatilization of oil components at temperatures much lower than their boiling points, hence protecting the integrity of thermally sensitive chemicals. A crucial aspect that determines the efficiency of steam distillation is the pretreatment of the plant material [8]. Pretreatment methods such as employing fresh (wet), cooled, or dried samples increase the moisture content, rupture cell membranes, and affect the ease with which the oil is freed from the plant tissue. However, there remains a paucity of evidence addressing the ideal pretreatment conditions for Eucalyptus oil extraction, especially when comparing species like *Eucalyptus camaldulensis* and *Eucalyptus torelliana*.

Specifically, there is ambiguity regarding the impact of alternative pretreatment methods such as employing fresh (wet) leaves, freezing the leaves at low temperatures (–4 °C), or drying the leaves on the total yield and quality of the extracted oil. Understanding these impacts is vital for optimizing the extraction process and the possible commercialization of Eucalyptus oil production nationally.

Hence the main aim of this study is to examine how different pretreatment types affect the production and quality of essential oils extracted from two *Eucalyptus species Eucalyptus camaldulensis* and *Eucalyptus torelliana* using steam distillation. The specific objectives are as follows: To study the effect of different pretreatment methods on the yield and quality of essential oils obtained from different Nigerian-based *Eucalyptus species*. The outcome of this study has important implications both scientifically and commercially. Scientifically, the research contributes to a deeper understanding of how pretreatment methods affect the chemical composition and yield of Eucalyptus oil which are valuable for further refinement of extraction techniques. This could serve as a basis for additional research into the optimization of essential oil extraction from other plant species. Commercially, identifying the most efficient pretreatment method will help reduce production costs, enhance product quality, and promote the use of locally sourced raw materials. This could encourage the establishment of small-scale extraction industries, create job opportunities, and reduce the country's reliance on imported essential oils.

2.MATERIALS AND METHODS

2.1 Materials

The leaves of *Eucalyptus camaldulensis* (Fig. 1a) and *Eucalyptus torelliana* (Fig. 1b) were collected from plantations at the Forest Research Institute (FRIN) in Ibadan, Nigeria and the Lagos State University Nigeria and identified at the herbarium of the FRIN. The leaves were collected early in the morning to ensure the samples were fresh and to minimize the loss of volatile compounds. Distilled water was used for the steam distillation process. n-Hexane reagent was obtained from Labio Scientific Centre, Lagos, Nigeria.

Fig. 1 ***Eucalyptus camadulensis*** **(a) and** ***Eucalyptus torelliana*** **(b).**

2.2Pretreatment procedure

Before extraction, the leaves of *Eucalyptus camaldulensis* and *Eucalyptus torelliana* were cut into smaller pieces of approximately 9.5 cm in length to increase the surface area and promote efficient oil release during the steam distillation process. The leaves were then pretreated using three different methods.

2.2.1Wet pretreatment

In the wet pretreatment, freshly harvested Eucalyptus leaves were washed with distilled water to remove any surface contaminants and then used directly in the extraction process without any further treatment. This method retains the natural moisture content of the leaves.

2.2.2Chilled pretreatment

For the chilled pretreatment, a portion of the collected leaves was stored in a freezer at –4 °C for 24 hours. The rationale behind this method is that the freezing process may cause cellular disruption, which in turn could facilitate the release of essential oils during distillation.

2.2.3Dry pretreatment

Another portion of the leaves was subjected to a drying process. The leaves were air-dried naturally for days until a constant weight was achieved. The drying process aimed to concentrate the oil content within the cells before extraction.

2.3Extraction procedure

Indirect steam distillation was used for the extraction of essential oils to prevent the breakdown of some components of the leaves. The indirect steam distillation apparatus (Fig. 2) was assembled on different retort stands. All connections between the round-bottom flask, biomass flask, adapters, condenser, and collection flask were clipped to ensure an airtight system. The pretreated Eucalyptus leaves were loaded into the biomass flask and distilled water was added to the steam flask and was sealed. Hot steam was generated by

heating the water at 100 °C. As the steam passed through the plant matrix, it caused the essential oil to volatilize. The oil-laden steam ascended into the condenser, where cold water was circulated to condense the steam into liquid. The condensed distillate, comprising a mixture of water and essential oil, was collected in the receiving flask. Using a separating funnel, the oil was separated from the water based on differences in density. The yield of the extracted oil was then calculated using Equation 1.

Fig. 2 Indirect steam distillation apparatus.

$$Percentage\ Yield = \left(\frac{W_2 - W_1}{W_3}\right) \times 100 \quad (1)$$

Where:

W_1 = Weight of the empty collection flask (in grams)

W_2 = Weight of the collection flask with the extracted oil (in grams)

W_3 = Weight of the initial plant material (in grams)

2.4 Analysis

To determine the chemical composition of the extracted oils, samples were analyzed using a gas chromatography-mass spectrometry (GC-MS) system (GC/MS-QP2010 Ultra) equipped with a TR-5 MS capillary column (30 m length, 0.25 mm internal diameter, 0.25 µm film thickness). Helium was used as the carrier gas with a flow rate of 1.21 mL/min. The oven temperature was programmed to increase from 100 °C to 260 °C at a rate of 10 °C/min, and the injection volume was set at 5 µL. The mass spectra were collected over a range of 10–850 m/z. A small aliquot of the essential oil was dissolved in n-hexane. The solution was then filtered to remove any particulate matter before injection into the GC-MS. The chromatograms were analyzed using the Wiley spectral library search program. Peaks corresponding to various compounds were identified based on their retention times, initial times, final times, peak areas, and height percentages.

3. RESULTS AND DISCUSSION

3.1 Yield of essential oils

The yield of essential oils for each experimental run as a function of extraction time, pretreatment type, and species type is presented in Fig 3 and 4. In general, the yield varied with the extraction time, pretreatment type, and species with wet and chilled pretreatments producing noticeably different results. For *Eucalyptus camaldulensis*, yields of the dried pretreated leaves varied from around 0.89–0.93%. Notably, the wet pretreatment leaves produced yields close to 1.40%. For *Eucalyptus torelliana*, the yields ranged from approximately 1.10% to 1.73% across various runs. The highest yield was observed for the chilled pretreated leaves for the *Eucalyptus camaldulensis* (1.4% at 135 min) and *Eucalyptus torelliana* (1.73%, 224.25 min) with *Eucalyptus torelliana* showing a higher yield at a longer extraction time.

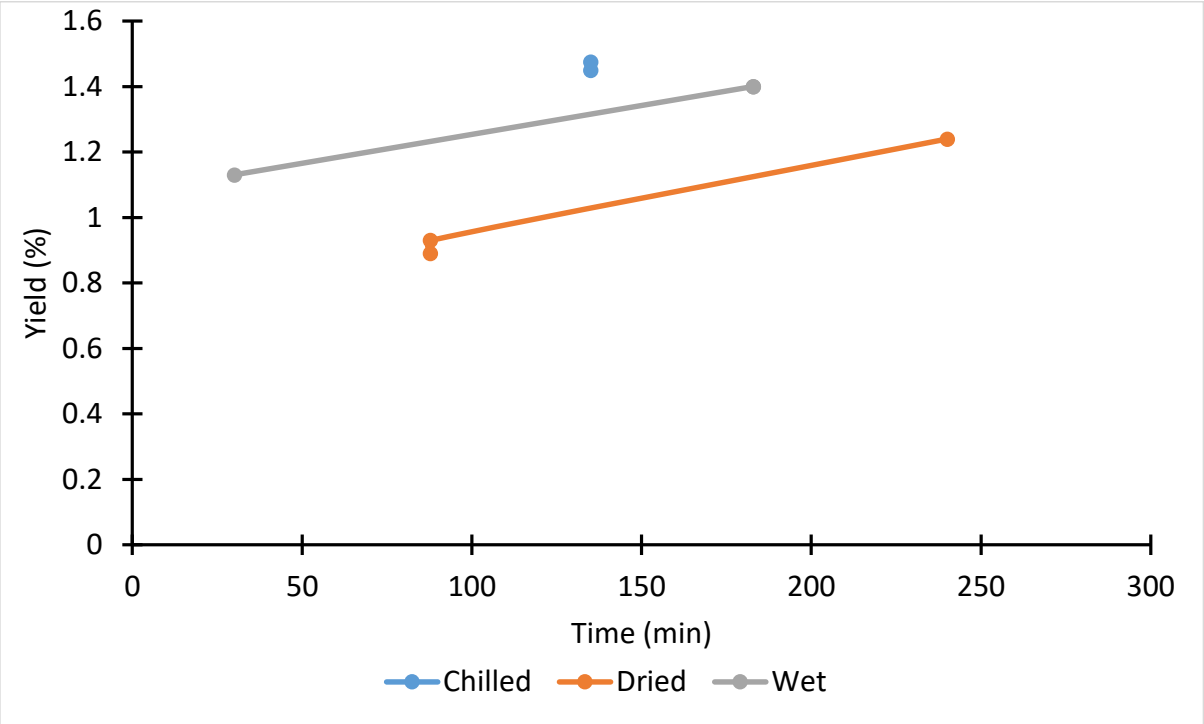

Fig. 3 Yield of essential oil for the different pretreatment types at different times for *Eucalyptus camadulensis*

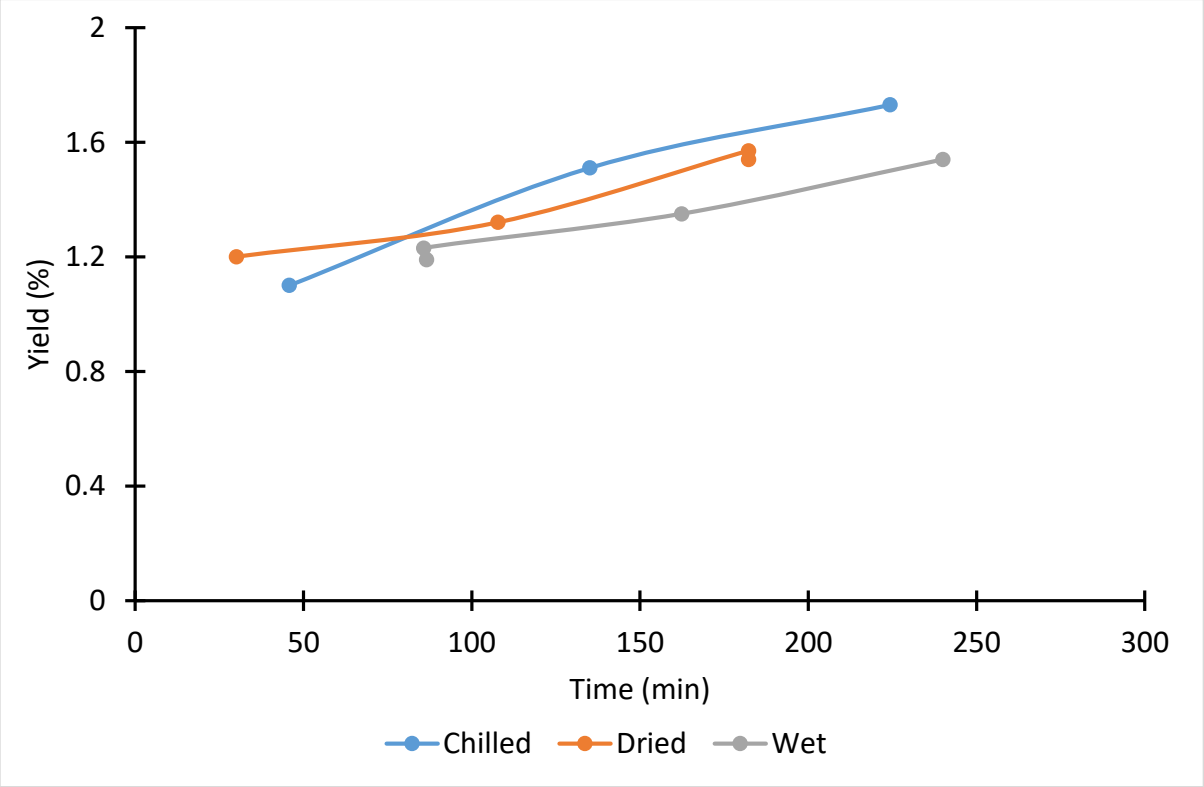

Fig. 4 Yield of essential oil for the different pretreatment types at different times for *Eucalyptus torelliana*

3.2Physical properties of the extracted oils

The physical properties of the essential oils were evaluated for both wet and dry extracts. The observations are recorded in Table 1 below.

Table 1: Physical Properties of Eucalyptus Oil

Property	Wet Extract	Dry Extract
Colour	Clear, colourless liquid	Clear, colourless liquid
Odour	Aromatic and camphoraceous	Aromatic and camphoraceous
Taste	Pungent, camphoraceous taste	Pungent, camphoraceous taste
Solubility	Insoluble in water but miscible with organic solvents	Insoluble in water but miscible with organic solvents
Density	0.872 – 0.897	0.872 – 0.897

The similarity in physical properties between the wet and dry extracts suggests that the pretreatment method does not significantly alter the fundamental characteristics of the oil. However, it is the chemical composition, as determined by GC-MS that provides deeper insights into potential differences.

3.3Chemical Composition of extracted oils

Results of the GC-MS analysis on two sets of samples from the wet extract of *Eucalyptus camaldulensis* (Table 2) and the other from the dry extract of *Eucalyptus torelliana* (Table 3). The detailed tables below list the compounds identified, along with their retention times, peak areas, and corresponding percentages.

Table 2: Chemical Properties of wet *Eucalyptus camaldulensis*

Peak	R. Time (min)	I. Time (min)	F. Time (min)	Area (%)	Height (%)	Compound Identification
1	6.226	6.167	6.400	0.14	0.15	Tetraethylene glycol
2	6.932	6.875	6.975	1.60	5.26	Eucalyptol
3	7.002	6.975	7.200	0.99	2.37	Eucalyptol
4	8.050	8.000	8.292	0.44	0.49	–
5	8.375	8.308	8.400	0.21	0.69	–
6	8.417	8.400	8.517	0.16	0.55	–
7	8.719	8.650	8.817	0.49	1.01	Isoborneol
8	9.020	8.925	9.225	0.97	1.45	α-Terpineol
9	12.538	12.483	12.875	1.26	1.18	Tetraethylene glycol
10	15.138	15.058	15.542	7.29	12.34	Pentaethylene glycol
11	16.805	16.758	16.883	0.13	0.66	n-Hexadecanoic acid
12	17.350	17.317	17.383	0.03	0.16	Ethene, (2-ethoxy-1-methoxyethoxy)-
13	17.571	17.392	18.608	16.26	16.23	Hexaethylene glycol
14	18.317	18.233	18.525	0.13	0.17	cis-Vaccenic acid
15	20.335	20.042	20.642	25.26	18.03	1,4,7,10,13,16-Hexaoxacyclooctadecane
16	21.425	21.342	21.667	0.23	0.27	1,4,7,10,13,16-Hexaoxacyclooctadecane
17	21.717	21.667	21.775	0.15	0.37	Floxuridine
18	21.900	21.775	22.075	0.39	0.33	Octadecanoic acid, 2,3-bis(acetyloxy)propyl ester
19	22.172	22.075	22.275	0.37	0.54	Octadecanoic acid, 2,3-bis(acetyloxy)propyl ester
20	22.375	22.275	22.500	0.89	0.81	2-Hydroxyethoxyethoxy
21	22.788	22.500	23.342	27.50	19.58	2-Hydroxyethoxyethoxy
22	24.992	24.642	24.992	15.10	17.35	Hexane, 1,2,3-trimethoxy-

Table 3: Chemical properties of dry *Eucalyptus torelliana* extract

Peak	R. Time (min)	I. Time (min)	F. Time (min)	Area (%)	Height (%)	Compound Identification
1	12.713	12.583	13.625	1.59	0.45	Tetraethylene glycol
2	15.078	15.025	15.492	5.37	7.52	Pentaethylene glycol
3	16.466	16.425	16.542	0.16	0.80	Hexadecanoic acid, methyl ester
4	16.793	16.758	16.892	0.45	1.89	n-Hexadecanoic acid
5	17.497	17.383	18.025	13.74	14.39	Heptaethylene glycol
6	18.173	18.125	18.208	0.15	0.71	Methyl stearate
7	18.300	18.267	18.417	0.40	1.22	Hexadecenoic acid, Z-11-
8	18.541	18.483	18.642	0.48	1.75	Octadecanoic acid
9	19.718	19.667	19.792	0.67	2.67	Methyl 12-hydroxy-9-octadecenoate
10	20.251	20.017	20.700	24.25	16.45	Heptaethylene glycol
11	21.747	21.650	21.958	4.14	10.48	Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl)
12	22.716	22.467	23.008	26.38	18.26	1,4,7,10,13,16-Hexaoxacyclooctadecane
13	23.233	23.167	23.308	0.08	0.13	9-Octadecenoic acid (Z)-, 2-hydroxy-1-(hydro...
14	23.432	23.308	23.533	3.17	8.33	Octadecanoic acid, 2,3-dihydroxypropyl ester
15	23.625	23.533	23.700	0.13	0.14	[2-(2-Hydroxyethoxy)ethoxy]
16	24.953	24.717	24.992	18.85	14.82	1,4,7,10,13,16-Hexaoxacyclooctadecane

3.4 DISCUSSION

3.4.1Effect of pretreatment on yield

The experimental results indicate that the pretreatment method significantly influences the quantity of oil extracted. Overall, wet pretreatment which preserves the natural moisture content of the leaves often produced yields that were either comparable to or slightly higher than those achieved with chilled or drying pretreatments. This suggests that the presence of intrinsic moisture may facilitate the vaporization of essential oil constituents during steam distillation. In contrast, while the chilled pretreatment (which disrupts cell structure via freezing) enhanced oil release, it did not consistently outperform the wet method. Drying pretreatment, on the other hand, led to the loss of some volatile compounds, thereby reducing the overall yield in certain runs.

The variability in yields can be attributed to several factors which include the water present in fresh leaves likely aids in the co-distillation of volatile oil components [9]. Freezing (chilled pretreatment) disrupts cell walls, facilitating oil release; however, excessive freezing may also cause partial degradation [10]. Drying exposes the leaves to prolonged heat, potentially leading to the degradation of sensitive compounds [11].

3.4.2Physical properties and their implications

The physical properties recorded in Table 1 for both wet and dry extracts show that the overall appearance, odour, taste, solubility, and density remain consistent despite differences in pretreatment. This uniformity is important from an industrial standpoint as it indicates that the extraction process does not compromise the intrinsic characteristics of the oil. The clear, colourless liquid with a characteristic aromatic and camphoraceous odour is highly desirable for both medicinal and cosmetic applications. The consistency in density (0.872 – 0.897 g/cm^3) further confirms the reproducibility of the extraction process.

3.4.3Chemical composition and bioactive constituents

The GC-MS analysis (Tables 2 and 3) indicates that the essential oils comprise a complex combination of chemical components. Notably, chemicals like eucalyptol (1, 8-cineole) are found in high quantities, and this compound is well-known for its antibacterial and anti-inflammatory activities. Other notable chemicals found include tetraethylene glycol, pentaethylene glycol, n-hexadecanoic acid, methyl stearate, and numerous cyclic ethers. The relative percentages of these components provide insights regarding the possible usefulness and stability of the oil in various applications.

For the wet Eucalyptus camaldulensis extract, the dominance of chemicals such as eucalyptol and the existence of numerous glycol derivatives imply that the oil has both medicinal and industrial use. In the dry *Eucalyptus torelliana* extract, a similar pattern is found, albeit the exact concentration of each constituent varies. These variances may have ramifications for the oil's usefulness in different end uses, such as in medications and cosmetics.

The precise chemical profiles obtained by GC-MS enable for the identification of minor chemicals that, despite their

modest concentrations, may exert synergistic effects on the oil's overall bioactivity. For instance, the presence of fatty acids and their methyl esters (such as n-hexadecanoic acid and methyl stearate) may add to the oil's antioxidant effects. Additionally, cyclic molecules like 1, 4, 7, 10, 13, 16-hexaoxacyclooctadecane present in considerable levels could have a role in the oil's stability and its capacity to operate as a natural preservative.

3.4.4Comparison with literature

In comparison to prior studies, the yields and chemical compositions identified in this research correspond with the established advantages of Eucalyptus oil. Research indicates that Eucalyptus oil generally comprises 67–97% 1,8-cineole [12]; notwithstanding variations in our study's specific percentages, the reliable identification of eucalyptol highlights the efficacy of the extraction method. Furthermore, the discrepancies noted between the two species and various pretreatment techniques highlight the necessity for a customized strategy in industrial applications. Pretreatment consistently produces a higher concentration of bioactive chemicals, scaling up the technique with chilled leaves may provide substantial economic benefits.

4. CONCLUSION AND RECOMMENDATION

This study indicates that pretreatment methods significantly influence the yield and chemical composition of Eucalyptus essential oil derived from *Eucalyptus camaldulensis* and *Eucalyptus torelliana*. Freshly harvested leaves subjected to chilled pretreatment yielded higher quantities of oil compared to wet or dried samples due to the disruption of the cell walls, facilitating oil release. While both species produced oils with consistent physical properties characterized as clear, colourless liquids with aromatic, camphoraceous odours subtle compositional differences were observed. Gas chromatography-mass spectrometry (GC-MS) analysis confirmed the presence of key bioactive compounds, including eucalyptol, glycol derivatives, and fatty acid esters, which underpin the oil's therapeutic and industrial value. These findings highlight the feasibility of establishing localized, small-scale extraction plants in Nigeria to reduce reliance on imported essential oils, stimulate agricultural value chains, and advance industrial sustainability.

Moreover, it is recommended that other pretreatment approaches be studied. Alternative approaches such as ultrasound-assisted extraction or microwave pretreatment may produce greater disruption of cellular structures, thus accelerating the release of essential oils. Such approaches hold the potential to dramatically improve extraction efficiency and overall oil yield compared to current techniques.

To encourage commercial use, it is vital to conduct pilot-scale studies that validate the laboratory findings under industrial circumstances and also provide significant insights into process economics, which are critical for successful industrial applications.

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