

Abstract

Optimal conditions for hydrodistillation were determined as an extraction time of approximately 60 minutes, a NaCl concentration of 2%, and a water-to-material ratio of 1:15 g/mL, yielding a maximum of 2.19%. For steam distillation, the optimal parameters were 500g of material, an extraction time of about 45 minutes at an evaporation rate of 0.13 ml/s, resulting in a highest yield of 1.57%. In micro-assisted hydrodistillation, the best conditions included an extraction time of approximately 30 minutes, a water-to-material ratio of 1:4 g/mL, and an extraction power of 380W, achieving a maximum yield of 1.4%.

GC-MS analysis identified 70 components, constituting 99% of the total composition. The primary compounds in black pepper essential oil were β -caryophyllene, 3-carene, D-limonene, β -pinene, and α -phellandrene.

Introduction

The black pepper plant, also known as "black gold" or "king of spices," is the world's most widely used spice due to its commercial, economic, nutritional, and medicinal value. Black pepper essential oil is also extensively employed in the food, beverage, and cosmetics industries. The essential oil's composition includes α -Pinene, β -pinene, δ -3-Carene, Limonene, E-Caryophyllene, and other compounds.

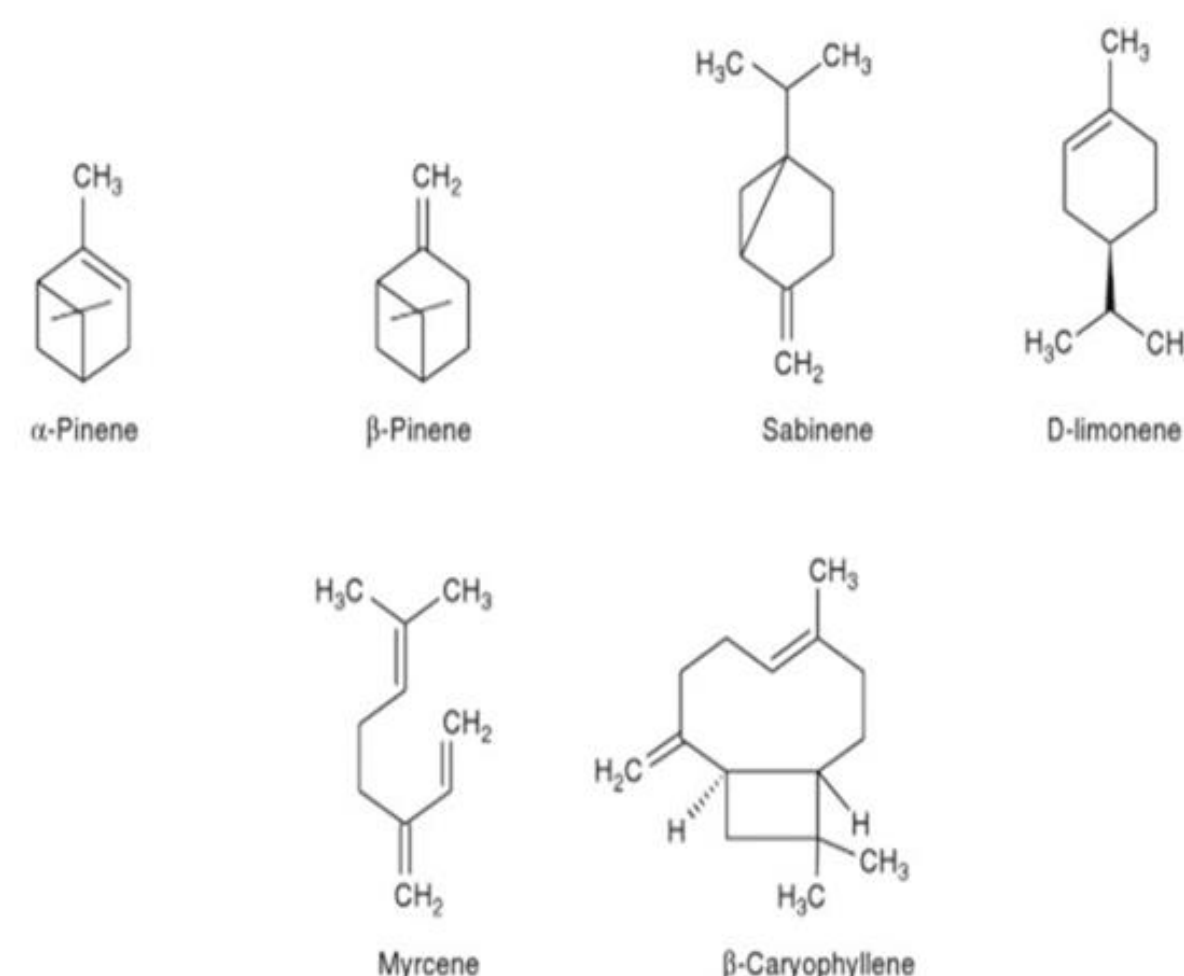


Figure 1. Structure of active substances

Methods and Materials

The materials used in this study were black pepper (*Piper nigrum* L.) seeds, cultivated, harvested, and processed in Chau Duc, Ba Ria Province. The black pepper seeds were initially picked and dried before undergoing a grinding process. The ground material was subsequently added to the distillation flask.

The extraction time commenced with the first drop of condensed essential oil collecting in the oil extraction system. Upon completion of the extraction process, the essential oil was recovered and dehydrated using sodium sulfate.

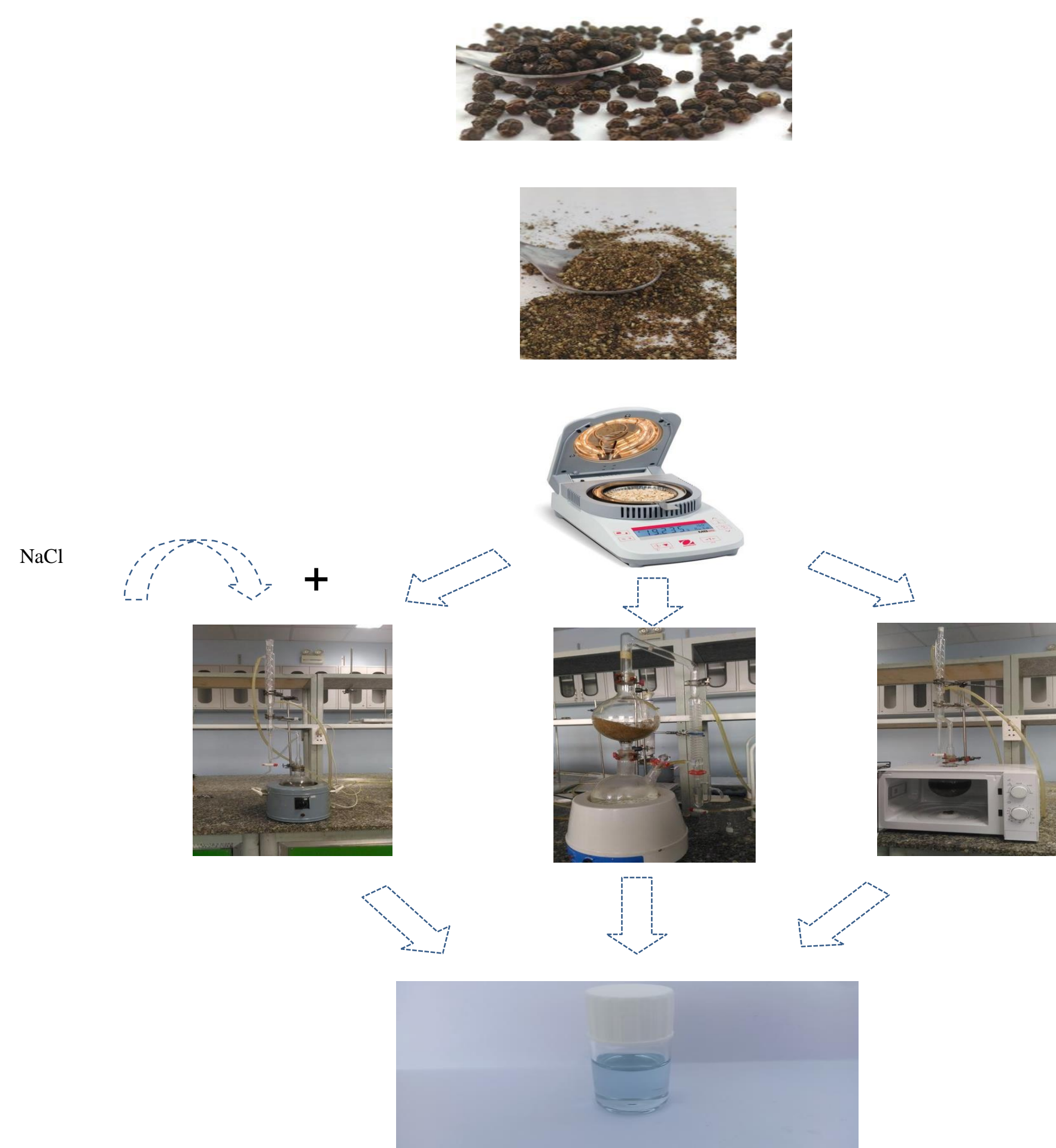


Figure 2. Experimental process

Results

Hydro distillation:

The ratio of raw material to solvent was varied (1/10, 1/15, 1/20, 1/25). Results indicated that a ratio of 1/15 yielded the highest oil content at 2.18%.

The extraction yield was found to plateau after 60 minutes, showing no significant increase with longer extraction times. However, the yield of black pepper essential oil increased as the extraction time was extended.

Steam distillation:

At an evaporation rate of 0.13 ml/s, the highest oil yield of 1.16% was achieved. This is attributed to energy efficiency and the oil's green color, suggesting minimal denaturation of components within the product.

The extraction yield stopped increasing after 45 minutes of extraction time.

The results also demonstrated the influence of material quantity on essential oil yield. A material amount of 500g resulted in the highest oil yield of 1.57%.

Microwave-assited hydrodistillation:

Results indicate that a 1/4 (g/mL) ratio could significantly reduce water and material consumption. Using excessive water during extraction can lead to increased oil diffusion into the water, causing essential oil loss and consequently lower yields.

A power of 540W produced the highest oil yield at 1.33%, but the essential oil had a dark yellow color due to a small amount of material splashing into the flask and burning. Consequently, 380W was selected as the power for subsequent experiments. The extraction yield stopped increasing after 30 minutes of extraction time.

Table 1. Composition of essential oils from different methods

Compounds	Methods		
	HD	SD	MD
α -Pinene	5.11	4.67	0.529
β -pinene	8.13	7.93	0.011
δ -3-Carene	17.12	15.49	2.544
Limonene	15.51	15.24	13.163
Caryophyllene	24.34	29.86	25.694
α -Copaene	2.67	4.36	0.052
β -Myrcene	2.60	2.06	0.065
α -Phellandrene	5.37	4.23	1.047
Terpinolene	1.03	0.82	0.195
α -Humulene	2.48	3.03	1.810

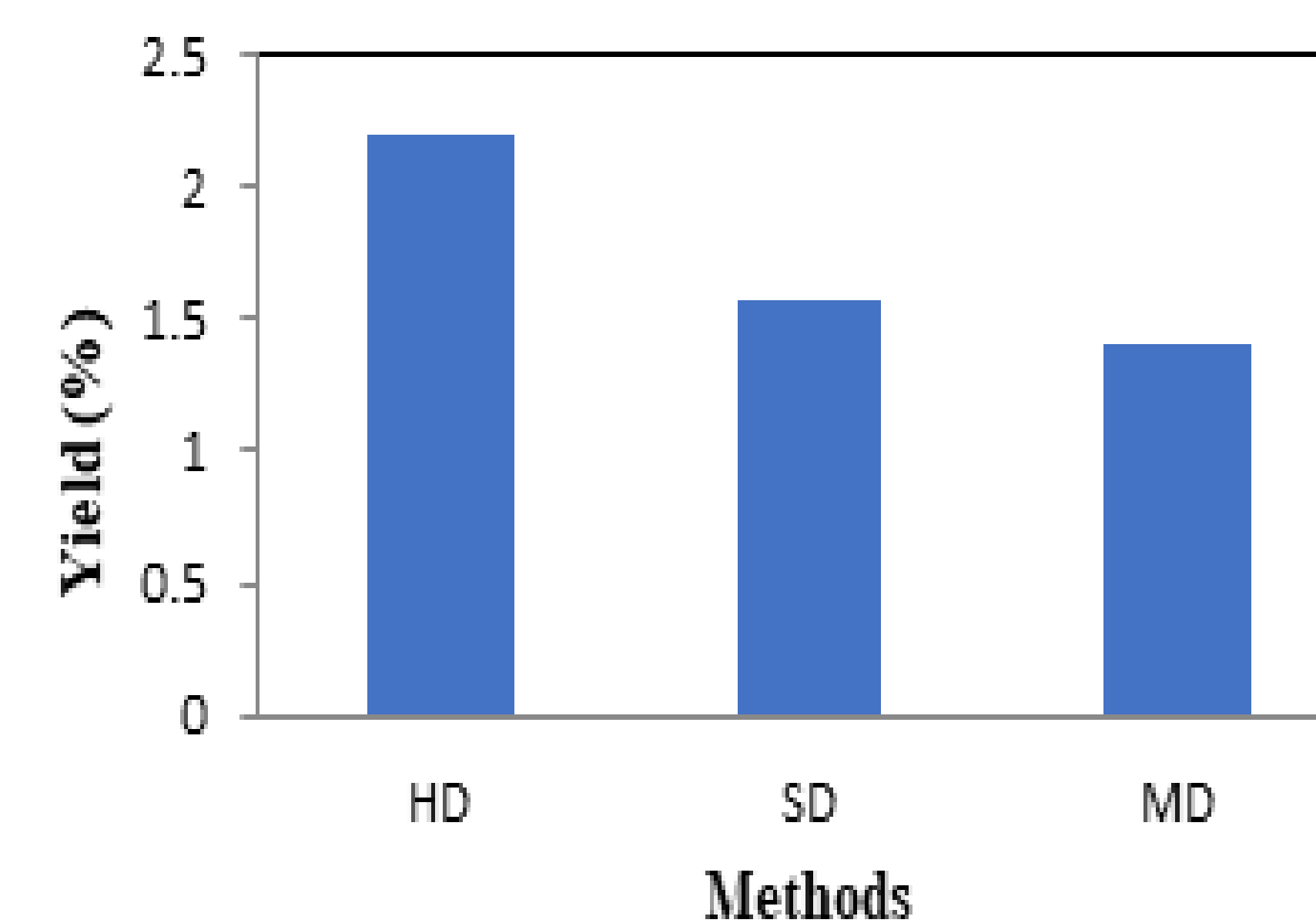


Chart 1. Compare the yields of different methods

Discussion

Chart 1 presents the yields of the distillation methods under optimal laboratory conditions. While all three methods share similar influencing factors, the HD method stands out as more efficient. This is attributed to its immersion technique using NaCl as an electrolyte, which enhances water density and polarity. This modification facilitates the separation of essential oils from water, resulting in higher yields.

Conclusions

This study investigated optimal conditions for extracting essential oil from Vietnamese black pepper (*Piper nigrum* L.) seeds from Ba Ria Province using several laboratory-scale methods. GC-MS analysis determined the chemical composition of the produced oil. Hydrodistillation yielded 2.18% oil after approximately 60-90 minutes using a water-to-material ratio of 1:15 g/mL. Steam distillation produced 1.57% oil from 500g of material in 60-90 minutes at 190°C. Micro-distillation yielded 1.17% oil in 30 minutes with a water-to-material ratio of 1:1 g/mL and a power of 380W. GC-MS analysis identified α -Pinene, β -pinene, δ -3-Carene, Limonene, and E-Caryophyllene as components of the essential oil.

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