

STEAM DISTILLATION WITH INDUCTION HEATING SYSTEM: ANALYSIS OF KAFFIR LIME OIL COMPOUND AND PRODUCTION YIELD AT VARIOUS TEMPERATURES

(Penyulingan Wap Dengan Sistem Pemanasan Aruhan: Analisis Komposisi Minyak Limau Purut Dan Hasil Pengeluaran Pada Suhu Pelbagai)

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Abstract

The steam temperature during the extraction process has a great influence on the essential oil quality. This study was conducted to analyze the compound of kaffir-lime oil during extracting at different steam temperature using GC-MS analysis. The extraction was carried out by using steam distillation based on induction heating system at different extraction temperature such as 90° C, 95° C and 100° C, the power of the induction heating system is fixed at 1.6kW. Increment of the steam temperature will increase the oil yield. In terms of oil composition, extraction at lower temperature resulted high concentration for four marker compounds of kaffir-lime oil which are α -pinene, sabinene, limonene, β -pinene.

Keywords: Kaffir-lime oil, GC-MS analysis, and steam distillation-based induction heating system

Abstrak

Suhu wap semasa proses mengekstrak mempunyai pengaruh yang besar bagi kualiti minyak pati. .Kajian ini dijalankan untuk menganalisis sebatian minyak limau purut semasa diekstrak pada suhu wap yang berbeza dengan menggunakan analisis GC-MS. Pengekstratan telah dijalankan dengan menggunakan wap penyulingan berasaskan sistem pemanasan aruhan pada suhu pengekstrakan yang berbeza iaitu pada 90° C, 95° C dan 100° C, kuasa sistem pemanasan aruhan adalah 1.6kW. Meningkatkan suhu wap akan menyebabkan peningkatan hasil minyak. Dari segi komposisi minyak, pengekstraktan pada suhu yang lebih rendah menyebabkan kepekatan yang tinggi terhadap empat kompaun utama minyak limau purut iaitu α -pinene, sabinena, limonene, β -pinene.

Kata kunci: minyak limau purut, analisis GC-MS dan penyulingan wap berasaskan system pemanasan aruhan

Introduction

Essential oil is mostly used for flavouring, as perfumes or as fragrance[1] in many applications. Initially, several significant parameters should be considered to contribute the output yield and quality of the production. Have been noted, the significant parameters concern during extraction process is temperature, extraction time, and extract material condition [2-7].

The steam distillation is the conventional method used to obtain the essential oil from plant material because cleanliness, cheap, simple, ease design to compare to others advance technique [8, 9]. Countless research has been dictated highlight the effect of temperature to the production of essential oil and temperature plays important role in the extraction process [2, 7, 10-15].

Therefore, for every single of extraction method have focussed on temperature extraction either is fixed[5, 13-18] or vary with temperature[2-4, 11, 19-21] and from the analysis shows temperature will affect the yield of essential oil[3, 5, 21, 22]. Based on that point, the temperature becomes important rule and selected as the process variable in this study. Currently, steam distillation mostly used in industries of extraction essential oil. By using the basic idea of steam distillation, many techniques are extending suppose to optimize the performance of steam distillation in the process of extraction. Although this method known always become of benchmarking technique to others technique[1-3, 7, 10, 11, 23], but this method is the most popular technique in industrial and research work due to low cost, cleanliness, high production yield, simple operating procedure[24].

There has several numbers of research efforts denoted in order to control the steam temperature of steam distillation system to offer high output yield essential oil [25-27]. In this case study, application of PID controller has always be more popular technique since it's simple, ease of design, and cost required relatively low[25]. Another advanced technique also derived in optimized the output response of steam temperature such as hybrid fuzzy- PD plus PID controller, and self tuning PID controller [26, 27].

High Efficient Steam Distillation System

The conventional steam distillation system is modified to achieve the higher performance during the extraction process. Conventionally, the heating element applies to the plant steam distillation are electrical heating or gasses. But in this developed plant, induction heating system is introduced as heating element to the extraction process. Selection of the induction heating system because of their working principle which is a high efficiency, cleanliness and safe compared to other heating element.

Figure 1 shows a sketch of steam distillation system with induction based and in Figure 2 show the block diagram of the experiment setup for this system.

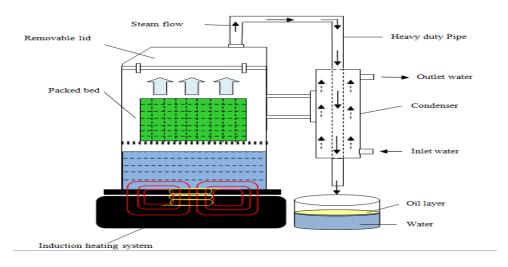


Figure 1. Distillation pot system prototype sketch

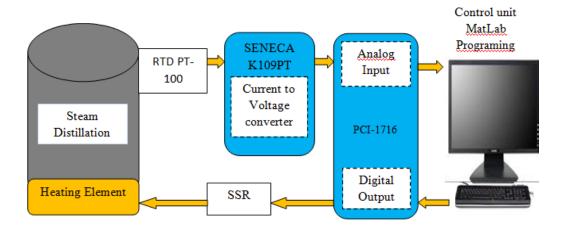


Figure 2. Experiment setup and system interface

The temperature of extraction will control by using Fuzzy plus P, I, and D proposed in [28] that give good performance which improve system response and uncertainties [29, 30].

Materials and Methods

Raw Materials

The extraction process starting with the preparation raw materials which are fruits of kaffir lime were collected at the mature stage from fruit orchards around Dengkil, Selangor. The essential oils of kaffir lime are obtaining used the kaffir lime peels because it had much of aromatic sense and more moisture compared to leaves of kaffir lime[31] which are contained high major volatile component such as limonone, citronellal and α - β pinane. Kaffir lime peels (350g) consistently are grounded to ensure the volatile compounds expose easily for each experiment running.

Extraction Procedures

The 350g ground material was stored in packed bed inside the steam distillation pot. Before the process run, 3000ml water at room temperature is adding into steam pot to produce a steam during the extraction process. The steam produce will become as transportation medium of volatile compound and enhance the production yield of essential oil. The steam produced will bring the volatile compound through condenser before condensed and become a layer of oil and hydrosol. The hydrosol produced had follow isolation process where the sample was transferred to separator funnel and dichloromethane was added to separate between oil and liquid. After funnel was shaking, the oil layer appeared at top and liquid at bottom layer.

In this conducted work, induction heating system used 1.2kW to boil the water inside the steam pot until the steam temperature achieves the set point such as $90^{\circ}\text{C},95^{\circ}\text{C}$ and 100°C . The control strategies and technique used to control the extraction temperature as introduced in the previous work[28]. The extraction time is stopped after 2 hours because not much produces essential oil when it approximately achieves saturated extraction point and avoid energy waste. The quantity of oil yield will be determined based on follower equation.

$$yield\% = \frac{Weight\ of\ extract\ recovered\ (kg)}{Weight\ of\ fresh\ kaffir\ limr\ peel\ (kg)} \times 100$$

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Chromatographic Analysis

The separation of essential oil volatile compound was performed on a Hewlett-Packed gas chromatograph with flame ionization detector using a fused silica capillary column HP5-MS ($30m \times 0.25mm$ internal diameter with $0.25\mu m$ film thickness). The injector temperature was $250^{\circ}C$. The oven was set $60^{\circ}C$ for three minutes and temperature was programmed to increase from $60^{\circ}C$ to $200^{\circ}C$ at a rate of $6^{\circ}C/min$ and from $200^{\circ}C$ to $325^{\circ}C$ at a rate of $30^{\circ}C/min$. The carrier gas was helium at a flow rate of 1.0mL/min, and the sample volume injected was 0.1 ml of the pure oil [10].

Identification of essential oil compounds was based on comparison of the mass spectra obtained in the gas chromatography with those of compounds registered in the NIST MS (National Institute Standard and Technologies Mass Spectra of the standard Wiley Register of Mass Spectra), library database of the GC-MS. The chromatography was performed on a gas chromatograph (Model Agilent 7890), using a fused silica capillary column HP5-MS, coupled to a selective mass detector (Model 5975C). The detector temperature was 280° C, and the ionization mode was electron impact at 70Ev [10].

Results and Discussion

The experiment of extraction kaffir-lime oil is conducted to see and evaluate the quantity and quality of the output yield by applying the control scheme towards the process. In this work, the oil yield of kaffir-lime was found out and volatile compound of the kaffir-lime has been identified. The volatile compound is identified through GC-MS and GC analysis provided.

The effect of temperature on production yield of kaffir-lime essential oil

In the experiment of extraction kaffir lime essential oil, the yield of oil produced is monitored by taking a reading of oil yield for every 15 minutes. Eight different tubes are used within 2 hour extraction process to separate the oil produces every 15 minutes. The layer of oil appears on top of separator tube with physical oil is clear liquid appearance. The oil that appears on top easily to suctioned and stored in the bottle. Yield of essential oil is calculated based on a wet weight basis since the drying process is escaped during pre-process the raw material preparation. The experiment was repeated three times for every different temperature The oil yield accumulated in percentage during the extraction process at different temperature is tabulated in Table 1 below/.

Experiment	90 °C	95 °C	100 °C
Yield1(%)	1.738	3.249	3.517
Yield2(%)	1.652	3.114	3.544
Yield3(%)	1.822	3.331	3.445
Mean of yield(%)	1.737	3.231	3.502
Standard deviation of yield(%)	0.069	0.089	0.042

Table 1. Oil yield of Kaffir lime with different temperature parameter

From the result, the oil yield accumulated along the extraction process is increased when the temperature is increased. For temperature control at 90°C, the yield is not impressed when the steam is controlled at low temperature by giving yield equal 1.737%. When the temperature is controlled at 95°C, the yield is increased to 3.231%. A last experiment conducted without control element and give yield equal 3.502%. It shows that the yield

of essential oil is proportional to the temperature. From the standard deviation, a small standard deviation of oil yield indicates that the percentage yield for every experiment have values that are very close to the mean value.

Table 2. Kaffir-lime oil compound from GCMS analysis

	Compounds	Retention Index (RI)	Relative Peak Area (%)		
No			90 °C	95°C	Uncontrolled (100 °C)
1	α-Thujene	0924	0.072	0.158	0.105
2	α-Pinene	0932	3.223	3.109	2.959
3	Sabinene	0969	48.5	46.165	46.349
4	β-Pinene	0974	10.088	9.321	8.759
6	Myrcene	0988	1.469	1.446	1.338
6	δ-2-carene	1001	-	0.057	-
7	Limonene	1024	27.714	26.446	25.742
8	γ-terpinene	1054	0.053	0.190	0.168
9	Terpinolene	1086	0.095	0.079	0.042
10	Linalool	1095	0.028	0.687	0.781
11	Citronellal	1148	3.247	6.633	7.146
12	Terpinen-4-ol	1174	0.457	1.054	0.888
13	α-terpineol	1186	0.163	0.438	0.473
14	Citronellol	1223	-	0.097	0.435
15	Citronellyl acetate	1350	0.117	-	0.222
16	α- copaene	1374	0.092	0.365	0.302
17	β- сораепа		-		0.309
18	Gemacrene D	1484	0.185	0.206	0.035
19	δ- cadinane	1513	0.081	0.266	0.233

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The effect of temperature control on quality of kaffir-lime essential oil

This analysis is carried out to identify the volatile compound in kaffir-lime oil during extracted in different temperature control parameters. This part of the analysis consists of Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometry (GC-MS) test toward the experimental sample.

Five marker compounds that fanned out from this analysis have marked for each GCMS result. Data GCMS shows that the main compound in kaffir-lime oil is appeared for all experimental samples. The marker compounds in kaffir-lime oil are α-pinene, sabinene, β-pinene, limonene and citronellal.

Based on two tests conducted, nineteen volatile compounds are identified as tabulated in Table 2. The compound that derives from the kaffir-lime oil start with the compound α -thujene at retention index 0924 and finish by δ -cadinene at retention index 1513. In kaffir-lime oil, there have five marker compounds that contribute to the odour, colour and flavour which are α -pinene, Sabinene, β -pinene, limonene and citronellal varies in between retention index from 0932 until 1148.

During the temperature control at 90°C, the coefficient of marker compound is relative high except citronellal. For uncontrolled temperature parameter, coefficient of marker compound shows it relative low except the fifth compound which is citronellal contribute high coefficient. In kaffir-lime oil, the quality oil depends on the existing of marker compound that have high coefficient. But, high coefficient of citronellal will affect the odour of kaffir-lime oil with rancid smell. Overall, the quality of kaffir-lime oil is better when the temperature parameter is control compared without temperature control

Conclusion

From the experiment conducted, it can be concluded that by increasing the steam temperature, the oil yield has also been increased. However this increment is not always a good indicator since some of the undesired compound i.e citronelal has also increased. By controlling the steam at lower temperature, it produced high value kaffir-lime marker compound such as α -pinene, sabinene, β -pinene and limonene as compared to the oil without temperature control. It can also be noticed that by applying temperature control technique, citronelal compound can be reduced.

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