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EFFECT OF TEMPERATURE ON KAFFIR LIME OIL BY USING HYDRO-DIFFUSION STEAM DISTILLATION SYSTEM

(Kesan Suhu Ke Atas Minyak Limau Purut Dengan Menggunakan Sistem Penyulingan Penyebaran Hidro)

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Abstract

Extraction temperature is one of an important parameter that can give large effect on output yield and quality of essential oils. For that reason, temperature is chosen as a process variable in this study. Fruit of kaffir limes was collected from orchards around Dengkil area around January 2013. Fresh peels of kaffir lime undergo weighing process. A 0.35 kg grounded kaffir lime peels for each steam temperature setting which are 80 ^oC, 90 ^oC, 95 ^oC and uncontrolled temperature (100 ^oC). The steam temperature setting was achieved by using a HFPID controller scheme by controlling the voltage fed to the heater. The proposed research applied the concept of hydro diffusion where steam and extract (water and essential oil) move naturally downwards by earth gravity downwards to the condenser. The hydro diffusion steam distillation plant enhances oil recovery by minimizing oil waste in boiling water and its opposite from the conventional steam extraction where steam goes upward. From the results, it can be concluded that different temperature will give different effects on the quality and quantity of essential oil. For each steam temperature setting, it produces a different composition of the constituents.

Keywords: essential oil, steam distillation system, hydro-diffusion

Abstrak

Suhu pengekstrakan adalah salah satu parameter penting yang boleh memberi kesan yang besar ke atas hasil pengeluaran dan kualiti minyak pati. Atas sebab itu, suhu telah dipilih sebagai proses pembolehubah dalam kajian ini. Buah limau purut telah dikutip dari kebun di sekitar kawasan Dengkil sekitar Januari 2013. Kulit segar limau purut menjalani proses timbang berat. Sebanyak 0.35 kg kulit limau purut yang telah dikisar untuk setiap tetapan suhu wap iaitu 80 ^oC, 90 ^oC, 95 ^oC dan suhu yang tidak terkawal (100 ^oC). Tetapan suhu wap telah dicapai dengan menggunakan satu skim pengawal HFPID dengan mengawal voltan yang disalurkan kepada pemanas. Penyelidikan ini menggunakan konsep penyebaran hidro dimana wap dan pati (air dan minyak pati) bergerak secara semula jadi ke bawah oleh graviti bumi, ke bawah ke pemeluwap. Kaedah penyebaran hidro meningkatkan perolehan minyak dengan mengurangkan sisa minyak tertinggal dalam air mendidih dan bertentangan dari pengeluaran stim konvensional di mana wap pergi ke atas. Daripada keputusan, boleh dibuat kesimpulan bahawa suhu yang berbeza akan memberi kesan yang berbeza terhadap kualiti dan kuantiti minyak pati. Bagi setiap tetapan suhu wap, ia menghasilkan komposisi yang berbeza.

Kata kunci: minyak pati, sistem penyulingan wap, penyebaran hidro

Introduction

Essential oils sometimes called volatile oil are aromatic substances [1-3] represent a small friction of plant's composition which can be found mostly in leaves and flowers [4]. It is multi-component chemicals that will produce a product that is highly aromatic and more concentrated than the original plant [2]. Essential oils and extracts obtained from various botanical plants have recently gained a great popularity and science interest. Essential oils have a wide range of uses and promises various significant benefits to human and industrial such as in aromas and fragrances [5-7], medical activities [3, 8, 9], as insect repellents and pesticides [10-12], cosmetics [11, 13], as a natural additive for food flavoring [4, 14], pharmaceutical industries [1-3, 5, 11] according to the composition of the oil. The quality of essential oil can be identified based on main compound found in the substance via Gas Chromatography-Mass Spectrometry (GC-MS) analysis [10].

Kaffir lime from the Rutaceae family contains two types of essential oils, makrut leaf oil and makrut (fruit peel) oil [15]. Kaffir lime is a well known herb used in the industry and get high demand in commercialization such as aromatherapy, potential use in medicine, cosmetic industry in making anti-dandruff shampoo and in the food industry as an ingredient in processing chili paste [194-196]. The compounds in the kaffir lime peels such as α-pinene, camphene, β-pinene, sabinene, mycrene, limonene, ɣ-terpinene, terpinolene, trans-sabinene hydrate, copaene, linalool, citronellal, terpinen-4-ol, citronellol, geraniol and δ-cadinene with the refractive index (RI) is 1.4729 [16]. Fruit peels are more aromatic and moisture compared to leaves. It also contributes to oil production in steam distillation [10].

The selection of manipulated variable and process variable of a particular control system is very important because these parameters can significantly influence production yield and chemical composition of essential oil. There are some parameters under considerations such as temperature, pressure, flow rate, extraction time, particle size of extract material and raw state material (dry or natural) [17-20]. The parameters under study showed different influences on the volatile oil extracted [17]. Some previous studies on essential oils stressed that temperature, extraction time and extract material condition have the most significant effect on the production yield [18, 21, 22]. But, pressure and flow rate give less influence to output yield as compared to the temperature, extraction time and size of material [21, 23, 24]. Controlling the temperature of the extraction is important since a slight variation of a few degrees may result in big losses of oil as well as in oils with very different chemical characteristics, making them either efficient or not, according to the purpose they will serve [11].

Materials and Methods

Sample preparations

Fruit of kaffir limes were purchased from orchards around Dengkil area during mid of January 2013. The fresh peels of kaffir lime were put at room temperature around 23 ^oC and it was protected from direct light. Then, peels of kaffir lime undergo weighing process. A 0.35kg grounded kaffir lime peels for each steam temperature setting which are 80 ^oC, 90 ^oC, 95 ^oC, and uncontrolled temperature which is 100 ^oC . The steam temperature setting was achieved using hybrid fuzzy plus PID (HFPID) controller scheme by controlling the voltage fed to the heater via power controller.

The extraction method by using hydro-diffusion steam distillation system

The hydro-diffusion steam distillation essential oil extraction system developed in this work is intended for extracting the essential oil by steam distillation technique to improve the quality of output yield and get high quantity of essential oil as compared with the conventional steam distillation. Hydro-diffusion steam distillation essential oil extraction system is visualized as in Figure1 and installed in Distributed Control System Laboratory (DCS) in the University Technology Mara, Malaysia. It consists of two main part which is distillation column itself and material tray. Heating element in this system is electrical heater and was placed in the distillation column to boil the water in order to produce the steam. The distillation column and material tray were design using stainless steel material.

This work demonstrates a new method for extracting the essential oils. The proposed research applied the concept of hydro diffusion where steam and extract (water and essential oil) move naturally downwards by earth gravity downwards to the condenser. It's opposite from the conventional steam extraction where steam goes upward. By using conventional steam distillation, the steam is not fully used for extraction. As steam is passed through the material tray, it condenses in the first element before going to the next and so on, where it releases its enthalpy of vaporization. The steams that release their enthalpy have the probability to return back to the distillation column. This can be proved by examining the water in the distillation column. The hydro-diffusion steam distillation plant enhances oil recovery by minimizing oil waste in boiling water. Some literature reports that by using a conventional steam distillation process, residual oil dissolved in the water will cause an odor nuisance and waste of the valuable product in the water stream [18]. Redistilling or cohobating is the process to optimize the recovery of essential oil in the wastewater. But the process offers high cost mainly heating and energy costs [18]. Hydro-diffusion also can prevent pollution [25].

The distillation column with 10 litre water is heated by electrical heater to produce steam. By using the hydro-diffusion system, the kaffir lime peels were placed in the material tray inside hydro-diffusion steam distillation plant. Pressurized steam from the top of the plant and passed through the botanical materials to vaporize the volatile oils in the plant material. Material tray was attached to a condenser build from copper material and water oil separator. The steam and oil vapor mixture is then going down through the small tube to the condenser. The volatile components were collected in a container. The collected essential oils were separated from water by using sodium anhydrous acid and dichloromethane which acted as catalysts using Vacuum Rotavap. The genuine oil then stored in sealed containers under refrigeration prior to analysis. . Extraction was performed approximately 2 hours because not much essential oil produces over 2 hours and save energy consumption. A schematic diagram of the complete system setup is shown in Figure 2.

Figure 1. Hydro-diffusion steam distillation system

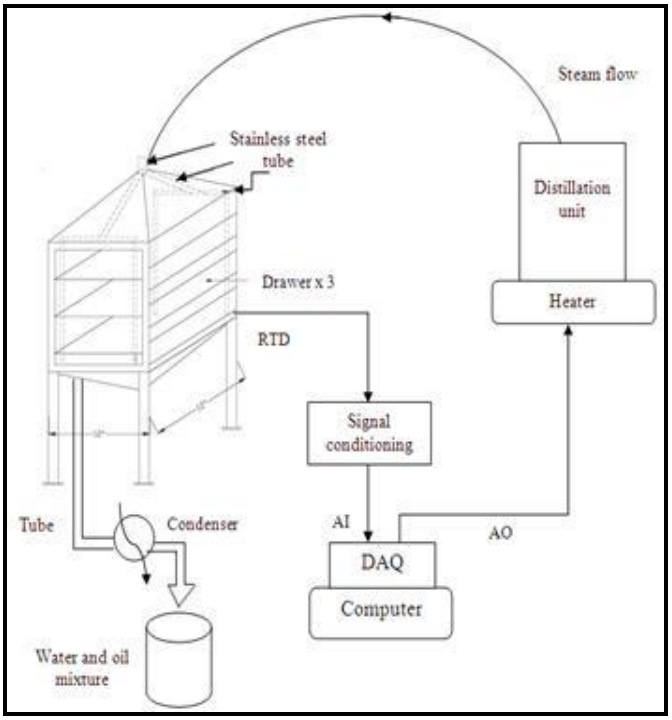

Figure 2. Schematic diagram of hydro-diffusion steam distillation system

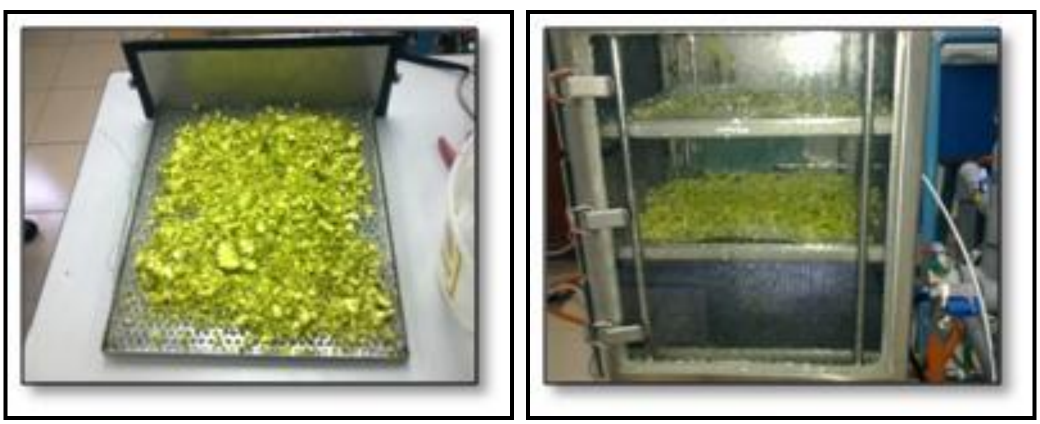

Figure 3. Sample preparations

The yield of the essential oil produced was estimated based on a wet weight basis. In order to measure the quantity of the collected oils, the percentage yields of essential oil were calculated as follows:

$$Yield\,(\%) = \frac{Weight\ of\ extract\ recover}{Weight\ of\ fresh\ citrus\ peels} \times 100$$

where the oil density of kaffir lime oil is 0.871g/mL [26]. So, the weight of extracting oil recovered in grams can be derived as:

$$Weight\ of\ extract\ recover\ (in\ gram) = volume\ of\ oil\ collected\ (mL) \times density\ of\ kaffir\ lime\ oil\left(\frac{0.871g}{mL}\right)$$

GC-MS conditions

Figure 4. GC-MS analysis

The composition of kaffir lime peel oil was tested by GC and GC/MS for qualitative analysis to identify active constituents. For GC analysis, a Shimadzu GC 2010 Gas Chromatograph which equipped with a FID detector and fused silica capillary column CBP5 (30m x 0.25mm, 0.25µm film thickness) was employed. The GC/MS analyses were carried out by using Agilent 5975C Mass Selective Detector equipped with an Agilent 7890A Gas Chromatograph. A HP-5MS fused silica capillary column (30m x 0.25mm) with 0.25µm film thickness was used. The carrier gas for GC is He with flow rate of 1ml/min and temperature programming as tabulated in Table 1 below:

Table 1. Setting for GC-MS analysis

Injector temperature	250 ^{o}C
Detector temperature	280 ^{o}C, with mass range recorder from 45-450 mass-to ratios, with electron energy of 70eV.
Oven temperature	The column oven was set to 60 ^{o}C for 3 minutes, and then the temperature was gradually increased up to 230 ^{o}C at a rate 3^{o}C/min. For 230 ^{o}C till 325 ^{o}C, the rate was maintained at 30 ^{o}C/min.

Identification of the chemical compounds

The identification of the chemical compounds was done by matching their mass spectra with existing data in the HPCH2205.L, Wiley7 Nist05.L and NIST05a.L library search data. Their retention indices were compared with Adam, 2007 library. Then, the major compounds in the kaffir lime peel were compared with those reported in the literature.

Results and Discussion

Comparative analysis of the effect of temperature on the production rate of Kaffir Lime peel oil

Figure 5 presents the production yield of kaffir lime peel using hydro-diffusion steam distillation essential oil extraction system. The extraction process was performed for 2 hours. From the experiment, the production yield start produced approximately after 50 minutes. As shown in Figure 5, two layers form after extraction process where the top layer is hydrosol and water at the bottom of the container. The oils were collected for every 10 minutes with the objective to see the flow rate of production yield. From Figure 5, it is interesting to note that the yield of essential oil is rapidly produced at the early stage of extraction as shown in container 1. The yield in container 1 was collected after 50 minute process started. Another important finding was that the yields start decreasing as time increasing. As shown in container 5, very poor amount of yield was collected after 2 hours.

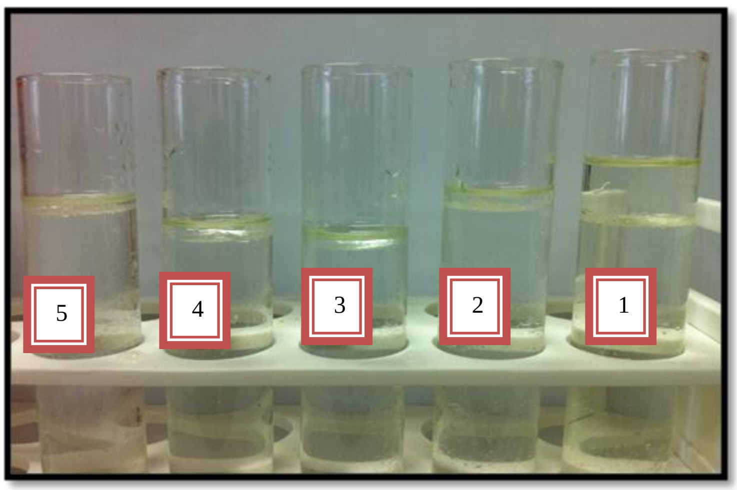

Figure 5. The production yield of kaffir lime peel

Table 2 tabulated the accumulated amount of percentage yield for each steam temperature control based on a wet weight basis. It means that no drying process involved during the sample preparation stage.

Table 2. Percentage of extracting kaffir lime essential oil

Steam temperature, (^oC)	**Production yield, (*mL*)**	**Percentage yield, (w/w)**
80	2.8	0.70
90	8.8	2.19
95	11.8	2.94
Uncontrolled	12.5	3.11

Referring to Table 2, by using hydro-diffusion steam distillation system for 2 hour extraction process, the percentage of kaffir lime oil yields at temperatures of 80 ^oC, 90 ^oC, 95 ^oC and uncontrolled temperature were 0.70% (w/w), 2.19% (w/w), 2.94% (w/w), and 3.11% (w/w), respectively. It was notable that the percentage yield increased as steam temperature increased. Uncontrolled steam temperature yielded the highest amount of essential oil. However, the observed difference of percentage yield between uncontrolled steam temperature and steam temperature at 95 ^oC is very small. The lowest yield was obtained at steam temperature of 80 ^oC. The most interesting finding was that the oil yields quite lucrative at 90 ^oC and showed 77.2% significant difference as compared to the oil yield at 80 ^oC. The graphical comparison of percentage yield at various steam temperature is shown in Figure 6.

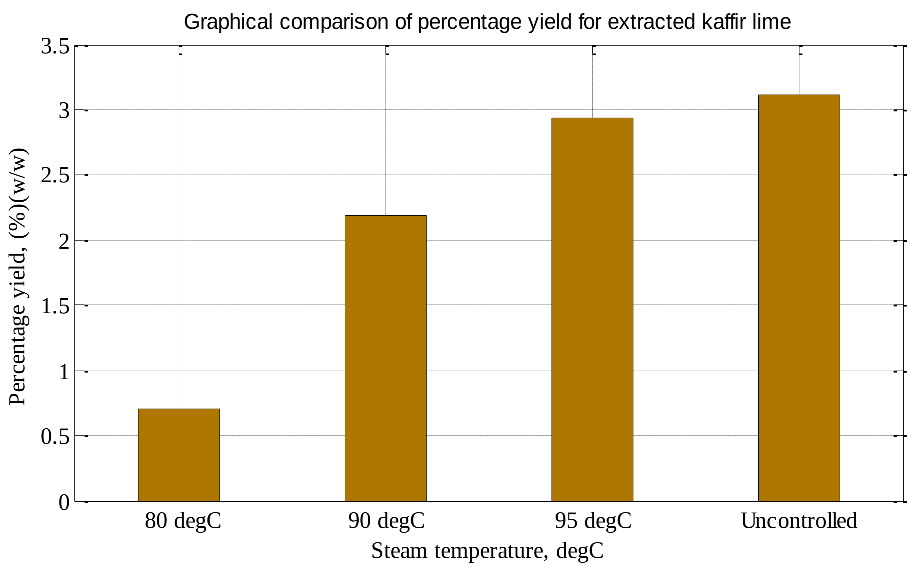

Figure 6. Graphical comparison of percentage yield for extracted kaffir lime peel using hydro-steam distillation system.

Figure 7 presents the production curves of kaffir lime oil yield at four different steam temperatures as a function of time where the oil yield was collected for every 10 minutes. The curves show that the extraction yield seems to have an asymptotic shape. It is apparent from this figure that oil yield was aggressively produced at an early stage of extraction process for all steam temperature settings. From the result, there are 3 different saturation times which are 60 min, 80 min and 90 min for a temperature of 80 ^oC, 90 ^oC, 95 ^oC and uncontrolled temperature, respectively. It was found that 80 ^oC has the fastest saturation time among all but produced the smallest oil yield. An extraction time of 60 min at 95 ^oC provide 3 times more yields as compared to those obtained by 80 ^oC.

Analysis of essential oil of kaffir lime by Gas Chromatography- Mass Spectrometry (GC-MS)

The analysis of essential oil from kaffir lime peels for each temperature was done by using Gas Chromatography-Mass Spectrometry (GC-MS). The chromatogram of GC-MS analysis for all temperature studies are presented in Figure 8 until Figure 11 for temperature 80 ^oC, 90 ^oC, 95 ^oC and uncontrolled temperature, respectively. The relative peak area in percentage (%) was calculated by comparing the peak ratio in chromatograms of GC and GC-MS. The compounds of essential oil obtained from kaffir lime peels from different temperature profile are summarized in Table 3. All the compounds were listed based on a retention index (RI).

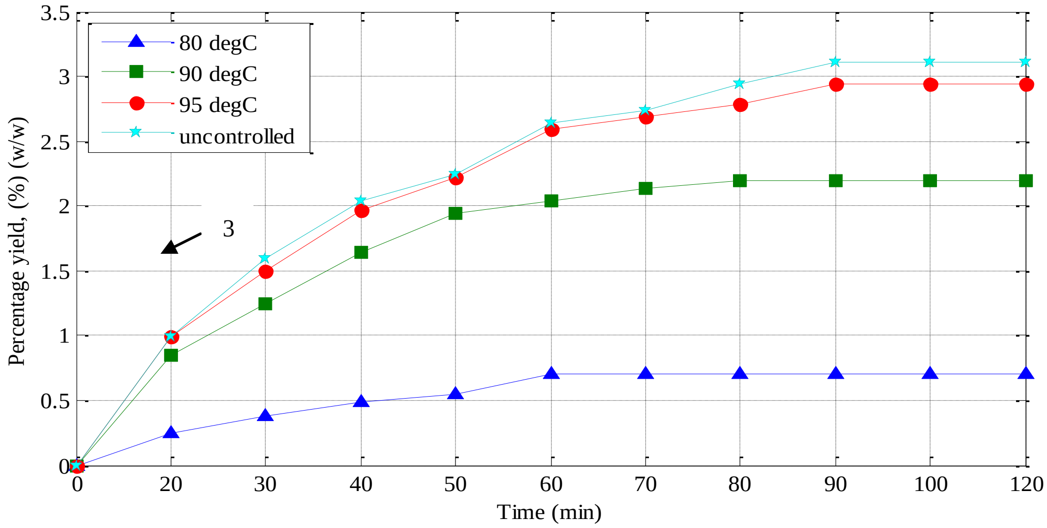

Figure 7. Production curves of kaffir lime oil yield at various steam temperature settings.

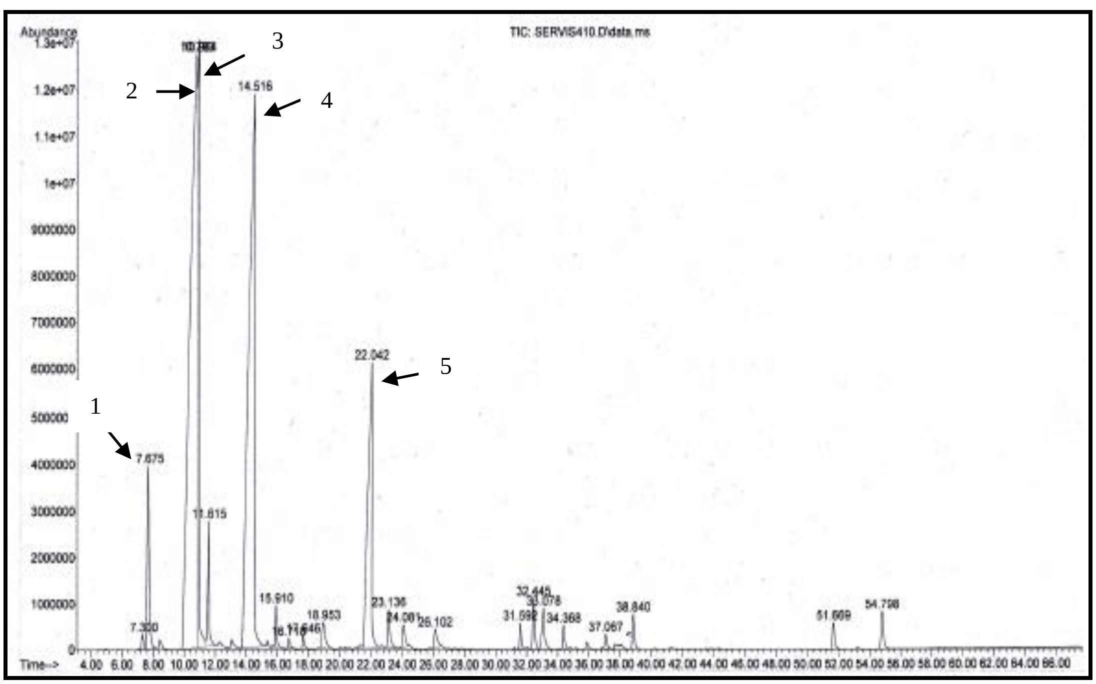

Figure 8. The GC-MS chromatogram of volatile compound from kaffir lime peel essential oil at 80 ^{o}C. The major compounds marked as (1) α-pinene, (2) Sabinene, (3) β-pinene, (4) Limonene, (5) Citronellal.

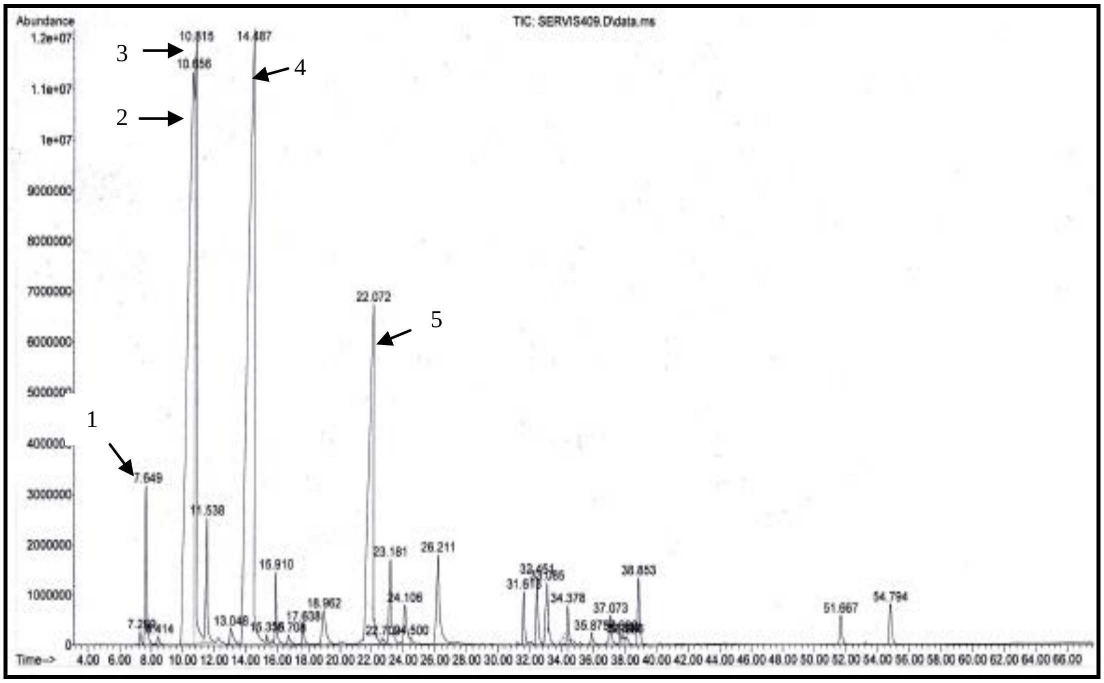

Figure 9. The GC-MS chromatogram of volatile compound from kaffir lime peel essential at 90 ^{o}C. The major compounds marked as (1) α-pinene, (2) Sabinene, (3) β-pinene, (4) Limonene, (5) Citronellal.

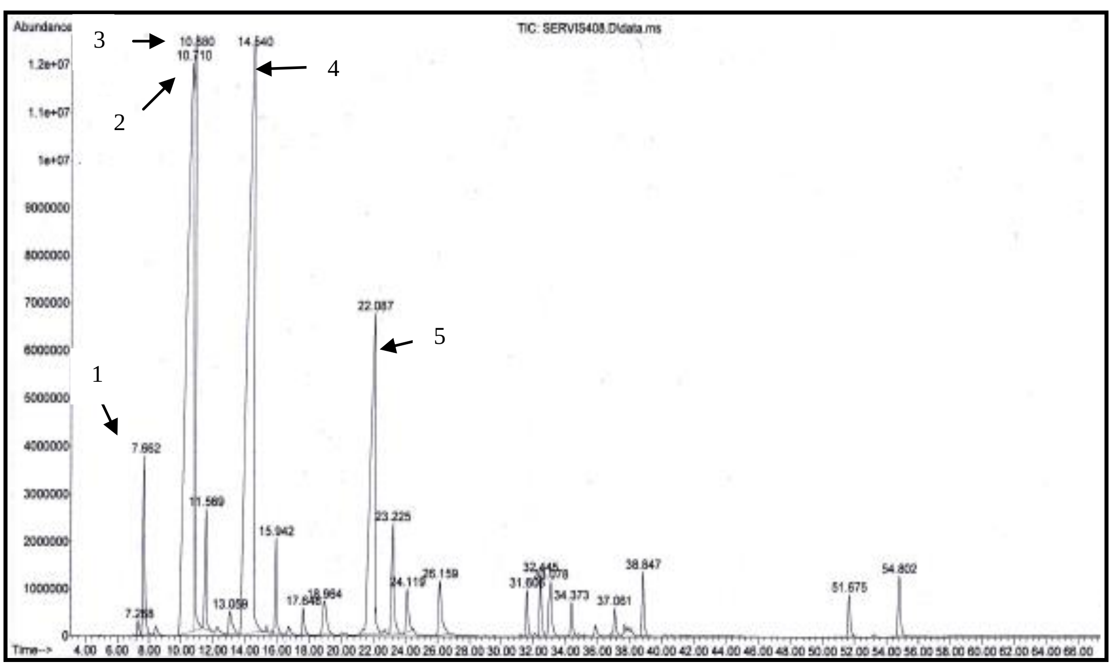

Figure 10. The GC-MS chromatogram of volatile compound from kaffir lime peel essential at 95 ^{o}C. The major compounds marked as (1) α-pinene, (2) Sabinene, (3) β-pinene, (4) Limonene, (5) Citronellal.

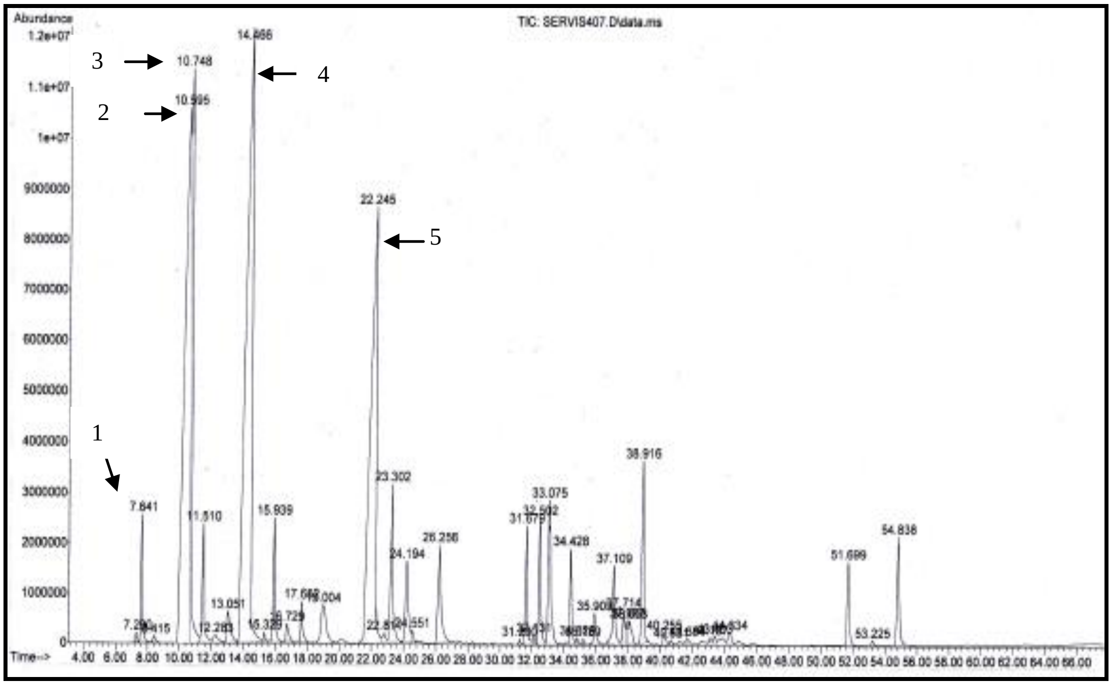

Figure 11. The GC-MS chromatogram of volatile compound from kaffir lime peel essential at uncontrolled temperature. The major compounds marked as (1) α-pinene, (2) Sabinene, (3) β-pinene, (4) Limonene, (5) Citronellal

It can be seen from the data in Table 3 that a total of 26 compounds was found in kaffir lime peels extracted using hydro-diffusion steam distillation system start with α-Thujene (0924) until δ-Cadinene (1522). Different numbers of compounds were extracted where a total of 19 compounds, 24 compounds, 19 compounds and 24 compounds were identified in temperature 80 ºC, 90 ºC and 95 ºC and uncontrolled temperature, accordingly. Therefore, the temperature at 90 ^{o}C and uncontrolled temperature can extract more chemical compounds. It is apparent from this table that 17 compounds appear in all temperature conditions such as α-Thujene, α-Pinene, Sabinene, β-Pinene, myrcene, δ-2-Carene, limonene, ɣ-Terpinene, linalool, citronellal, terpinene-4-ol, α-Terpineol, citronellol, α-Copaene, caryophyllene, citronellyl propanoate and germacrene D. All the major compounds of the kaffir lime peel oils such as α-pinene, sabinene, β-pinene, limonene and citronellal were detected for each temperature profile. Figure 12 presents the graphical major compounds in each temperature setting.

As refer to Figure 12, the major constituent of α-pinene at 80 ^{o}C, 90 ^{o}C, 95 ^{o}C and uncontrolled temperature were 2.515%, 1.806%, 2.207% and 1.255%, accordingly. The sabinene compound that indicates the spicy taste of the oil contributes the highest concentration among all with 45.594%, 36.394%, 37.481% and 27.498% at 80 ^{o}C, 90 ^{o}C, 95 ^{o}C and uncontrolled temperature, respectively. The third major compound is β-pinene which provide distinctive aroma where 8.974%, 8.597%, 8.714% and 7.147 were identified in respective temperature. Limonene that give a distinctive aroma of oranges provide 28.649%, 32.455%, 32.232% and 28.722% concentration at 80 ^{o}C, 90 ^{o}C, 95 ^{o}C and uncontrolled temperature, accordingly. Meanwhile, for citronellal compound which indicates the micro and insect repellent activity present 8.293%, 10.775%, 9.647% and 17.487% concentration. What is interesting in this data is that it is obvious for compound α-pinene, sabinene, β-pinene and limonene where the controlled temperature can extract more chemical compounds than uncontrolled temperature. The significant difference between these two conditions may due to oil experience thermal degradation which consequently losing some significant compounds in uncontrolled temperature. In contrast to earlier findings, citronellal was notably high in the oils collected from uncontrolled temperature. Figure 13 shows the color of the oils varies from light brown to dark brown as temperature increase. This finding supports previous research where different temperature will give an effect to the number of compounds extracted and quantity of essential oil [18, 21-22].

Table 3. Compound of essential oils obtained from the kaffir lime peel.

			Relative peak area (%)			
NO	**COMPOUNDS**	**RETENTION INDEX (RI)**	**80 ^oC**	**90 ^oC**	**95 ^oC**	**Uncontrolled temperature**
1	α-Thujene	0924	0.27	0.18	0.22	0.11
2	**α-Pinene**	**0932**	**2.515**	**1.806**	**2.207**	**1.255**
3	**Sabinene**	**0969**	**45.594**	**36.394**	**37.481**	**27.498**
4	**β-Pinene**	**0974**	**8.974**	**8.597**	**8.714**	**7.147**
5	Myrcene	0988	1.786	1.655	1.656	1.547
6	δ-2-Carene	1001	0.109	0.062	0.086	0.070
7	**Limonene**	**1024**	**28.649**	**32.455**	**32.232**	**28.722**
8	ɣ-Terpinene	1054	0.310	0.498	0.705	0.988
9	Terpinolene	1086	-	0.152	0.205	0.300
10	Trans-Sabinene hydrate	1098	0.146	0.136	-	0.273
11	Linalool	1095	0.541	0.810	0.802	0.921
12	**Citronellal**	**1148**	**8.293**	**10.775**	**9.647**	**17.487**
13	Terpinene-4-ol	1174	0.471	1.120	1.452	2.028
14	α-Terpineol	1186	0.350	0.583	0.668	1.242
15	Citronellol	1223	0.429	1.784	1.012	1.809
16	α-Copaene	1374	0.345	0.494	0.400	0.953
17	Caryophyllene	1417	0.280	0.313	0.258	0.725
18	Citronellyl propanoate	1444	0.185	0.391	0.315	0.909
19	Cis muurola-3, 5diene	1448	0.212	-	0.380	1.156
20	α-Humulene	1452	-	0.097	-	0.242
21	Geranyl propanoate	1476	-	-	-	0.810
22	Germacrene D	1484	0.091	0.430	0.162	0.517
23	β-Selinene	1489	-	0.032	-	-
24	Bicyclogermacrene	1500	-	0.073	-	0.256
25	α-Muurolene	1500	-	0.045	-	0.122
26	δ-Cadinene	1522	-	0.424	-	-
		TOTAL	**19**	**24**	**19**	**24**

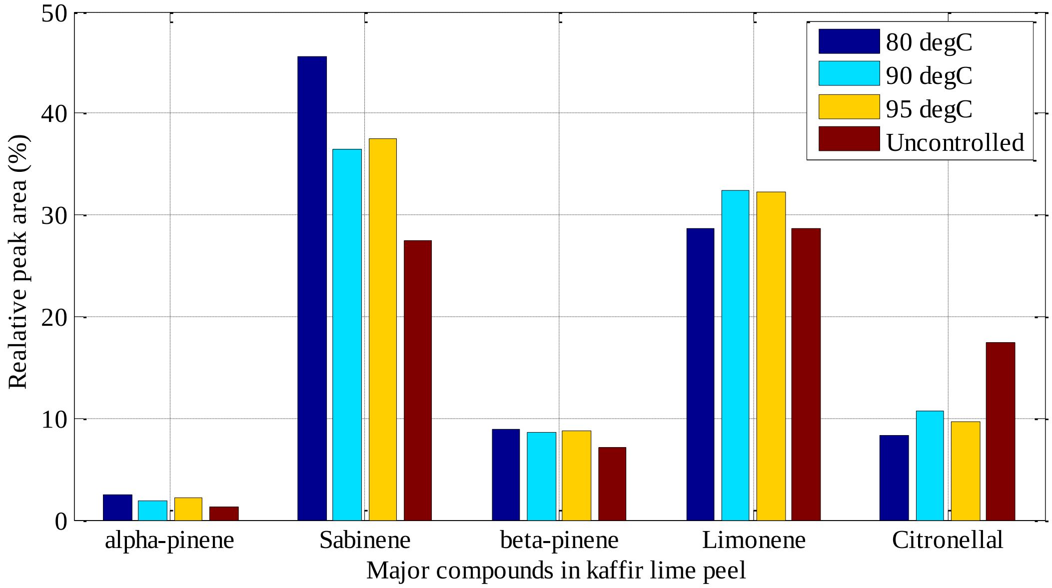

Figure 12. Major compounds in the kaffir lime peel

Figure 13. The pure essential oil from the kaffir lime peels

Conclusion

The comparative studies of production rate and quality of kaffir lime oil based on different steam temperatures have been studied. From the findings, it can be concluded that the percentage yield increased as steam temperature increased. Uncontrolled steam temperature yielded the highest amount of essential oil. However, the discrepancy between uncontrolled steam temperature and steam temperature at 95 °C is not obvious. A total of 26 compounds has been found in kaffir lime oil extracted using hydro-diffusion steam distillation system. The controlled temperature can extract more chemical compounds than uncontrolled temperature especially major compounds such as α-pinene, sabinene, β-pinene and limonene. Overall, the aims of this study to control the hydro-diffusion steam distillation system essential oil extraction system have been successfully achieved.

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