

Effect of Microwave Pre-Treatment for Hydrodistillation Extraction on Essential Oil Yield from Indigenous Herb, *Physalis Minima* Linn

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Abstract— Extraction of essential oil is a common process used to extract the phytochemical content from the plant that have beneficial impact health. *Physalis Minima* Linn is a medicinal plant that contains phytochemical that may beneficial to human. Hydrodistillation extraction technique has been used for extraction of oil from this plant by manipulating several parameter. As the objective of this study is to identify the phytochemical contains in the plant and to study the parameter that can affected the yield of extraction, three parameters had been varies which are extraction time (30min to 150min), pre-treatment power (180W to 540W) and pre-treatment time (30s to 120s). As the result from GC-MS analysis of phytochemical contains, it show that this plant contains aristolene, ibuprofen diethyl phthalate and cyclononasiloxane, octadecamethyl. Each of the phytochemical inhibits the nutritional value to human. The highest yield for this study is about 27.6% which parameter is effect of pre-treatment power at 540W while the lowest 4% at 30 minutes extraction time. Each parameter showed the trend that indicate the increasing of extraction yield. From GC-MS, four compound has been identified which are aristolene, ibuprofen, diethyl phthalate and octadecamethyl cyclononasiloxane.

Keywords— *Physalis Minima* Linn, phytochemical, extraction, health benefit, GC-MS

I. INTRODUCTION

According to the dictionary, essential oil is define as a natural oil typically obtained by distillation and having the characteristic odor of the plant or other source from which it is extracted. There are many studies related to the extraction of essential oil from many types of plant or sources. In this study, the plant use to extract the essential oil is *Physalis Minima* Linn. The function of the essential oil is define according to the bioactive contain in the essential oil. Each bioactive compound is carrying their unique ability to treat human. There several study conducted relating to extraction of essential oil from *Physalis Minima* Linn. To date, many researches regarding *Physalis Minima* were done for the study of phytochemical contains in the plant that can give some health benefits to human. There are several types of extraction methods likes microwave assisted extraction, soxhlet extraction,

supercritical fluid extraction and many others. In this study, type of extraction used is hydrodistillation extraction method.

Physalis Minima Linn or known as “Pokok Letup” in Malaysia is classified as medicinal plant in many regions and culture especially in India. As they believe this plant contains some bioactive compound or called as a phytochemical to treat some health problems likes gastric, stomach pain, constipations and some others^[1]. This plant used traditionally to treat the health problem^[1, 2]. Even though this plant is easily found, but not many people concern or realize the ability of this plant to treat health problem. The chemical composition exist in the seed is the oil and the protein. The oil contains the fatty acids, viz palmitic, stearic, oleic, linoleic and small amount of hexadecenoic and hydroxyl fatty acid, while the fruit of the plant contain 61.4% juice and 76.7% moistures. Besides that, the fruit also contain the sugar, tannins, mineral, protein and a good amount of vitamins. The ratio of vitamin to the juice is 24.45mg: 100ml juice^[1].

Table 1: The Characteristics of the *Physalis Minima* Linn Seeds

Characteristic of Seeds	<i>Physalis Minima</i>
Length (mm) ^a	3.5
Breadth (mm) ^a	2.6
Thickness (mm) ^a	1.1
wt of 100 seeds (g)	1.0
Moisture (%)	5.6
Oil (%) ^b	40.0
Protein (%) ^b	17.9
Ash (%) ^b	2.1
Crude fiber (%) ^b	24.2
Hull/ kernel ratio	30:70

(Source: *Characteristics and compositions of Carissa spinarum, Leucaena leucocephala and Physalis minima seeds and oils*)

^a Average of 10 seeds

^b Dry basis

According to the study done by^[3], the seeds of *Physalis Minima* Linn contain about 40% (based on the dry basis). The oil contents from the seed are about 40%. This plant also contains the protein and fiber. The crude fiber content in the plant is quite higher compare to another herbs that been study by T. Chandrasekhara, 1984 which is 24.2% compare to 21.6% for *Carissa spinarum* and 12.3% of *Leucaena leucocephala*. The protein contents in the seed of *Sunberry* are 17.9%.

Table 2: The Characteristic of the Extracted Oil from *Physalis Minima*

Characteristic of Oil	<i>Physalis Minima</i>
Specific gravity, 30/30°C	0.9176
Refractive index at 40°C	1.4679
Iodine value (Wijs)	122.5000
Acid Value	0.7000
Saponification value	189.0000
Unsaponification Value (%)	0.8000

(Source: Characteristics and compositions of *Carissa spinarum*, *Leucaena leucocephala* and *Physalis minima* seeds and oils)

The characteristics of the oil is represented in Table 2. The iodine value of the oils is 122.5 shows that this plant is a semidrying type [3]. Semidrying oil is oil that will partially harden when exposed to air. It will create a thin layer (film). The value of iodine is represent the amount of unsaturation matter in oil. The saponification value of the oil is 189 while the unsaponification value is 0.8%.

Table 3: The Fatty Acid Contents in *Physalis Minima*

The Fatty Acid contents	<i>Physalis Minima</i> (%)
Palmitic	10.5
Stearic	8.6
Oleic	17.3
Linoleic	61.4
Linolenic	0.0
Hexadecenoic	0.1
Epoxy	0.6
Hydroxy	1.5
Arachidic	ND ^d
Lignoceric	ND ^d

(Source: Characteristics and compositions of *Carissa spinarum*, *Leucaena leucocephala* and *Physalis minima* seeds and oils)

^dNot detected

The fatty acid contains in the oil is represented in Table 3. The highest fatty acid contains is linoleic type of fatty acid which is 61.4%. This type fatty acid is major in plant lipids and from dietary plant oil. The second higher value is the oleic fatty acid, 17.3%. This is the most common monoenic fatty acid in plants and animals [4]. The plant give a positive respond to the qualitative test for the epoxy and hydroxyl fatty acid, meanwhile through the Halphens Test, the cyclopropane fatty acid is absences in the oil extract [3].

The objective of this study is to identify the phytochemical that exist in the plant (*Physalis Minima* Linn) and to study the parameter that can affect the yield of extraction. In order to get the optimum yield, several parameter have been varies to get the result. As this plant is consider as a medicinal plant, but not many people concern about this plant, so the study is conducted to prove the content of the plant. The health benefits of the plant are as below:

Health Benefit of *Physalis Minima* Linn

As this plant is classified as a medicinal plant as it contains some nutritional properties and functional properties. Medicinal plant is define as a plant that at least one of the part from the plant (leaves, fruits, stem or root) can be used for the therapeutic purpose [5]. This plant contains the element that is a beneficial for some health problems [1] likes antibacterial [6], anti-malaria, anti-ulcer [7], anti-gonorrheal [8], anti-inflammatory [9], inhibitory effect on amylase, lipase and alpha glucosidase, hypoglycemic [10], cytotoxic [11], anti-lipid peroxidation and anti-fertility activity.

II. METHODOLOGY

A. Plant Materials

The plant material used in this study is *Physalis Minima* Linn. The sample plant was collected in Perak. For this study, the whole plant (fruits, leaves and stem) was being used. After the collection, the plant will be washed before it being dried naturally (not in a direct sunlight). After the plant has dried, it's being cut into the small size (0.3mm) for the experiment by using the cutter. In this study, the plant is being extracted by using the distilled water as a solvent.

Figure 1: *Physalis Minima* Linn

B. Microwave Pre-Treatment

The dried plant was weighted 10g and was placed in a flat bottom flask with the addition of 30ml distilled water to wet the sample, and then being pre-treat in the microwave for a power of 180W, 270W, 360W, 450W and 540W for a time of 30s, 60s, 90s, 120s, 150s and 180s. The temperature of the sample being monitored not more than 80°C to avoid the burning effect of the sample. For 360W, the expected time for pre-treatment was 30s, 60s and 90s only. Above 90s, the temperature of the sample was more than 80°C which will cause the burning effect to the sample. For power equal to 270W, the time was between 30s and 150s. After 150s, the temperature was higher than 80°C. After the pre-treatment time was finished, the sample was immediately put into a 2L beaker full of ice for a fast cooling process and the 50ml solvent (distilled water) was added immediately into the sample. The temperature of the sample was being monitored until it reached 40°C. When the temperature of the sample achieved 40°C, the sample will proceed to the Clevenger apparatus for the hydrodistillation process take placed.

C. Hydrodistillation

The hydrodistillation extraction was performed using Clevenger apparatus. In hydrodistillation procedure, the dried plant (10g) from the pre-treatment process with size of 0.3mm was extracted by distilled water in the flat bottom flask (500ml) that placed in Clevenger Hydrodistillation apparatus up to 120 minutes (2 hour). A major reason of hydrodistillation extraction technique had been chosen is that this process is the simplest type of extraction and commonly used for extraction process.

D. Effect of extraction time against yield

To study the effect of extraction time, 10g of sample was weighted and undergo the pre-treatment for 60s at power 450W. After done

the pre-treatment, the sample was added by 50mL of distilled water and immediately placed in a 2L beaker full with ice for the fast cooling process. When the temperature of the sample reached 40°C, the sample will be added with another 70mL of solvent (total of 120ml of solvent used) for hydrodistillation. The Clevenger apparatus was being set up and the experiment will be proceed for the desired period of time, which is in this study, it is varying from 30 min to 150 min (30, 60, 90, 120 and 150 min). The oil will be collected after the hydrodistillation time finished.

E. Effect of pre-treatment power against yield

To study the effect of pre-treatment power, 10g of sample was weighted and undergo the pre-treatment for 60s at microwave power that varies from 180W to 540W (180, 270, 360, 450 and 540W). After done the pre-treatment, the sample was added by 50mL of distilled water and immediately placed in a 2L beaker full with ice for the fast cooling process. When the temperature of the sample reached 40°C, the sample will be added with another 70mL of solvent (total of 120ml of solvent used) for hydrodistillation. The Clevenger apparatus was being set up and the experiment will be preceded for about 2 hour (120 min). The oil will be collected after the hydrodistillation time finished.

F. Effect of pre-treatment time against yield

To study the effect of pre-treatment time on the yield of essential oil, 10g of sample was weighted and undergo the pre-treatment for four different time which is set from 30s to 120s (30, 60, 90 and 120s) at power 450W. After done the pre-treatment, the sample was added by 50mL of distilled water and immediately placed in a 2L beaker full with ice for the fast cooling process. When the temperature of the sample reached 40°C, the sample will be added with another 70mL of solvent (total of 120ml of solvent used) for hydrodistillation. The Clevenger apparatus was being set up and the experiment will be proceeding for 2 hour. The oil will be collected after the hydrodistillation time finished.

After the experiment was done, the oil collected will be analyzed by using Gas Chromatography-Mass Spectrometer or known as GC-MS.

G. Gas Chromatography- Mass Spectrometer (GC-MS)

The oil extracted from the process was identified using gas-chromatograph (GC-MS) equipment model VARIAN with a column of Varian 240-MS with the specification of diameter is 0.32mm, column length is 30m and the column thickness is 0.50µm. the column should be kept in a constant flow of 1.73 ml/min and the injection volume of 0.5µl was employed with the split ratio of 10:1 and the temperature of 270°C for injector and 200°C for ion-source. The oven temperature was set from 40°C (isothermal for 2 min) with an increase of 8°C/min, to 150°C, then 8°C/min to 250°C, ending with 20min isothermal at 280°C. The gas carrier for this equipment was Helium gas.

III. RESULTS AND DISCUSSION

1. Temperature Profile for extraction of Physalis Minima Linn

Figure 2 show the temperature profile graph for this study. As can be seen, the first 40 min of the extraction, as the time increased, the temperature increase rapidly while after 40min, the temperature start to stable until the extraction time finished (120min). This

show that the highest temperature for this extraction process is below 100°C .

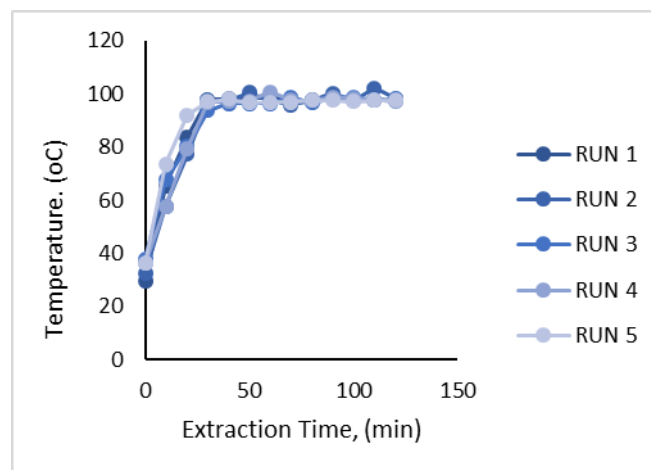


Figure 2: The Temperature Profile Graph

2. Essential Oil Yield

The percent yield of oil was calculated by using formula^[12]. Table 4 shows the percent of oil yield from the extraction on the parameter of effect of pre-treatment power while Table 5 shows the result of oil yield for the parameter effect of extraction time and Table 6 shows the result for parameter effect of pre-treatment time.

As can be seen, the oil yield increased for effect of pre-treatment power and effect of extraction time parameter, but showed the difference pattern for effect of pre-treatment time parameter. For effect of pre-treatment time, for 30s to 60s, the yield was increasing but start to decreased after 60s pre-treatment time. The highest oil yield was at effect of pre-treatment power, which is about 27.6% while the lowest at 4% yield at extraction time of 30min. the highest yield for effect of pre-treatment time is 21.3% which is at 60s.

The extraction time of 30min has the lowest yield due to the lowest extraction time. As the extraction time will affected the oil yield^[13]. As the time increased, the yield also increased. The pre-treatment process also affected the yield of oil extracted^[14, 15]. As the function of pre-treatment is to help in increasing the oil yield by breaking down the wall of the plant. The longer the pre-treatment time will affect the yield due to the tendency of the burning effect to the plant sample. So, for this study, the optimum pre-treatment time is about 60s.

Table 4: Oil Yield for Effect of Pre-Treatment Power

Microwave Power (W)	Weight of Beaker (g)	Weight of beaker + oil (g)	Weight of oil (g)	Yield (%)
180	332.22	334.17	1.95	19.50
270	332.45	334.52	2.07	20.70
360	332.53	334.68	2.15	21.50
450	332.26	334.71	2.45	24.50
540	332.67	335.43	2.76	27.60

Table 5: Oil Yield for Effect of Extraction Time

Extraction Time	Weight of	Weight of beaker +	Weight of oil (g)	Yield (%)

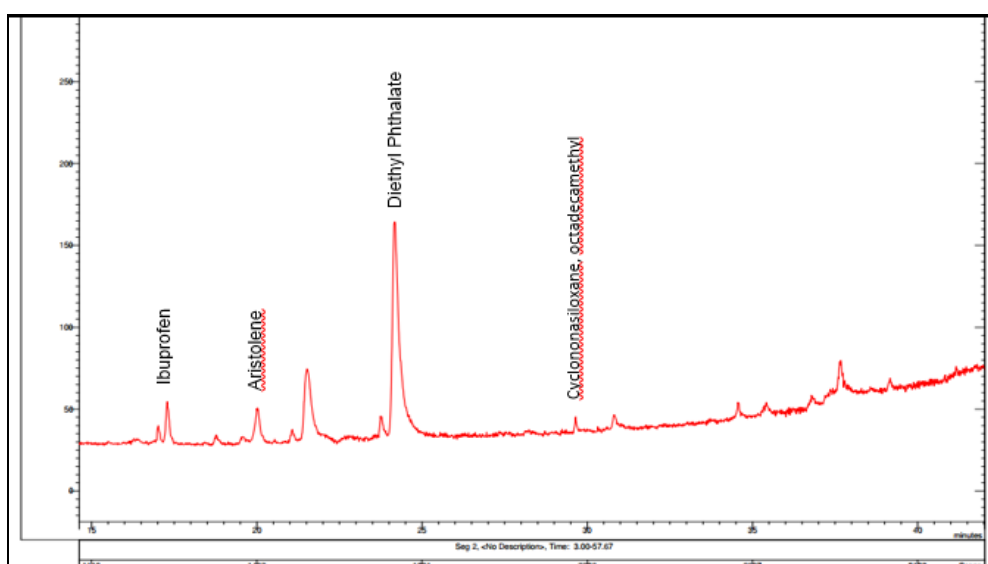
(min)	Beaker (g)	oil (g)		
30	332.61	333.01	0.40	4.00
60	332.42	333.68	1.26	12.60
90	332.68	334.47	1.79	17.90
120	332.55	334.97	2.42	24.20
150	332.28	334.72	2.44	24.40

Table 6: Oil yield for Effect of Pre-Treatment Time

Pre-Treatment Time (s)	Weight of Beaker (g)	Weight of beaker + oil (g)	Weight of oil (g)	Yield (%)
30	332.40	334.23	1.83	18.30
60	332.63	334.76	2.13	21.30
90	332.52	334.30	1.78	17.80
120	332.58	334.56	1.98	19.80

Gas Chromatography-Mass Spectrometer (GC-MS) Analysis

There are four compounds was identified in *Physalis Minima* Linn by GC-MS analysis. The compound that had been found with their retention time (RT), concentration (%) and group are presented at (Table 7 and Figure 3). The highest concentration (%) compound founded by GC-MS is Diethyl Phthalate (5.296%) followed by (-)-Aristolene (1.063%). The lowest concentration is Cyclononasiloxane, Octadecamethyl- (0.127%). Ibuprofen concentration is 0.994% and this compound exhibits the anti-inflammatory agents^[16]. Aristolene is a compound that carrying the toxicity agent to this plants^[17]. Diethyl phthalate and octadecamethyl cyclononasiloxane has been founded by the research done before this^[18]. Diethyl phthalate is a colourless liquid. This is the reason the oil extracted from this plant is colorless.

Figure 3: GC-MS analysis on *Physalis Minima* Linn ExtractTable 7: GC-MS Analysis on *Physalis Minima* Linn Extract

Peak	R. Time	Compound	%	Group	Health Benefits
1	17.2824	Ibuprofen	0.994	Carboxylic acid	Anti-Inflammatory ^[16]
2	20.0377	(-)-Aristolene	1.063	Terpene ^[17]	Cytotoxic ^[17]
3	24.1811	Diethyl Phthalate	5.296	Butyl ester ^[18]	Color and odor of oil
4	29.6463	Cyclononasiloxane, Octadecamethyl-	0.127	Benzene Methyl	-

I. CONCLUSION

Physalis Minima Linn have some phytochemical contents that carry the beneficial agents to human health likes ibuprofen and aristolene which act as anti-inflammatory and toxicity respectively. This will give the value to this plant to be commercialize. As the aim of this study is to identify the phytochemical and the parameter affect the extraction yield, so this study is conclude by each

parameter affect the extraction yield. The affected yield by the extraction time parameter. If the extraction time is small (30min) the yield is lower. The optimum extraction time for hydrodistillation is about 2 hours. The pre-treatment time also give the big impact to extraction yield.

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