Effect of drying temperature on the physical and chemical properties of water extract of *A.Malaccensis* leaves

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***Abstract*— *A.Malaccensis* have been proven by many researcher that there are alkaloids, tannins, saponins, flavonoids, terpenoids and phenolic compound in this species. The objective of this research are to study the effect of drying temperature on chemical and physical properties of *A.Malaccensis* using Far Infrared Radiation (FIR) dryer and to investigate the active compound extracted from hydrodistillation method. For the drying process, *A.Malaccensis* leaves were divided and dried at 40°C, 50°C and 60°C using FIR dryer and being extracted using hydrodistillation for 3-4 hours. The analysis on the physical properties of this 3 sample show that the amount of moisture content that have been evaporated during drying process were increase accordingly to the temperature such as 40°C:58.97%, 50°C:69.12% and 60°C:78.37%. Color of the leaves became darker (more brownish) as the temperature were increased up to 60°C. For chemical properties, there are two equipment that have been used which are Elemental Analyzer (EA) and Gas Chromatography-Mass Spectrometry (GC-MS). From EA analysis, it shows that CHN amount at 40°C were (45.5165: 5.8065: 3.089), at 50°C were (47.9018: 5.9076: 3.9693) and at 60°C were (47.9358: 5.8284: 3.8766). For GC-MS analysis, it shows that 48 compounds were found in sample A (40°C), 35 compounds in sample B (50°C) and 33 compounds in sample C (60°C). Result from this research show that drying temperature at 40°C have higher amount of compound extracted from the hydrodistillation method.**

Keywords*— *A.Malaccensis, drying temperature, FIR dryer, hydrodistillation, EA, GC-MS, physical properties, chemical properties.

I. INTRODUCTION

Nowadays, gaharu or agarwood are getting attention from researcher because of their advantage in health and medical industry. This agarwood are from Thymelaeceae family has been recorded more than 2000 years ago. It has been supplied from sources all around the world in Middle East and East Asia market. Long time ago, agarwood has been used in Indian and Chinese culture for an important treatment in their culture. Based on Indian Council, they conclude that agarwood is considered stimulant, antiasthmatic, carminative, tonic, aphrodisiac and astringent. It also can cured diarrhea, gout, rheumatism, liniment in various skin diseases and paralysis A.Malaccensis is a tree that have ability to produce resin in order to recover themselves from bacteria or wounded. There are many compound that can be found in this species such as phenolic compound, flavonoids, terpenoids, alkaloids saponins and many more. For example, this species is currently being researched for health applications [1] like alternatives herbs that can reduce fever because of this species has natural phenolic compound. The objective of this research are to study the effect of drying temperature on chemical and physical properties of A.Malaccensis using Far Infrared Radiation (FIR) dryer and to investigate the active compound extracted from hydrodistillation method. The extraction of active compound from *A.Malaccensis* leaves are very vital in order to get the best result. There are many type of drying equipment that can be used for drying process of this leaves such as conventional oven, microwave oven and far infrared radiation dryer. FIR dryer transfer heat through radiation and the optimization temperature during drying is between 40°C until 60°C. FIR dryer becoming an important source of heat treatment in the food industry because of advantages such as equipment compactness, fast transient response, significant energy saving, and easy accommodation with convective, conductive and microwave heating [2]. Far infrared radiation drying system generally consists of an insulated drying chamber, which is designed to withstand lower level of pressure; an infrared radiator (or infrared heater), which is used to supply thermal radiation to a drying product and a control system for the infrared radiator [3].

Extraction method is required in order to study the chemical and physical properties of this species, thus it is very important to choose the best extraction method for *A.Malaccensis* sample. There are many ways to extract compound from this species such as hydro distillation, solvent extraction (Soxhlet extraction), supercritical fluid extraction, solid phase micro extraction (SPME), steam distillation and many more. Many research found that different extraction method resulted in different compounds composition thus it can probably affect the pharmacological effect of this species in different way of extraction [4]. In this research, hydrodistillation method were choose as the extraction method for A.Malaccensis leaves.

In this study, there are two types of analysis that is used to determine the active compound in the sample which are Gas Chromatography-Mass Spectrometry (GS-MS) and Elemental analyzer. GC-MS analyzed chemical profiles of agarwood oil in order to know the classification of the oil according to their properties [5]. Meanwhile, carbon, hydrogen and nitrogen amount were analyze using Elemental Analyzer. For physical properties of water extract of this sample, the moisture content evaporated and color of the leaves was observed. For moisture content, weight of the leaves before dry and after dry is recorded [6].

II. METHODOLOGY

A. Sample Preparations

Species of *Aquilaria malaccensis* fresh leaves were collected from Jalan Kebun, Shah Alam. Damaged and diseased leaves were taken out from acquired samples. The leaves is then cleaned with clean water in order to remove any impurities as shown in Figure 1. The leaves that is rinsed by water is left at room temperature for 24 hours so that excess water can be removed [7].

Figure 1. *Aquilaria malaccensis* fresh leaves

A. Drying Procedure

The weight of the leaves was recorded before drying process in order to determine moisture content of the sample. The initial weight of the sample were recorded. The sample was separated into 3 part and were dried 6 hours using FIR dryer at temperature 40°C, 50°C and 60°C [8]. After drying process, weight of the leaves were recorded as shown in Figure 2 before grounded with Retsch SM100 into small pieces. The grounded leaves was kept inside covered plastic.

Figure 2: Weighted of leaves after drying

B. Hydrodistillation Extraction

Five grams of grounded leaves from each temperature were soaked with distilled water with ratio of (1:50) for two days before continued with hydrodistillation process. The extraction process was performed for 3-4 hours until the extraction completed. The extracted sample was collected in 250ml bottle and rotary vacuum evaporator were used to remove the excess water in the sample extraction [9]. After rotary evaporator, the sample was mixed with 5 ml of methanol before being analyzed using GC-MS. The remaining leaves from hydrodistillation were filtered and kept for Elemental Analyzer analysis.

C. Analysis of Physical Properties

After the drying process, color of leaves at different temperature was captured and observed. Moisture content was determined by calculated the difference between weight before and after drying. Before leaves were dried, weight of the leaves was recorded and after the drying process, the weight of leaves was recorded again [10].

Moisture content evaporated = [(A - B) / A] x 100%

*A = Before drying
B = After drying

D. Analysis of Chemical Properties

Compounds in the sample were determined by using Gas Chromatography – Mass Spectrometer (GC-MS) (Varian Inc.) installed with silica capillary column DB1 (J & W Scientific 30 m x 0.25 mm, 0.25 µm film thicknesses). The temperature of the column oven was set to 60°C to 230°C with an increment of 3°C/min. Oil was injected by using 50/100 µL syringe. Injector and detector temperature were set at 250°C and 280°C. Helium gas was used as the carrier with electron energy of 70eV and the flow rate of 52.5 mL/min. The retention time selected was 100 min [11]. The filtered samples of gaharu leaves from hydrodistillation process was analyzed using elemental analyzer. The sample for C/H/N analysis was weighed in tin capsule. The required amount is 12-15 mg. The tin capsule with sample was wrapped and placed in the autosampler.

III. RESULTS AND DISCUSSION

A. Effect of drying temperature on moisture content

Table 1 below show amount of moisture content evaporated at 40°C, 50°C and 60°C during the drying of *A.Malaccensis* leaves in FIR dryer. Temperature of 60°C has the highest moisture content that have been evaporated. This is because at the higher temperature, more heat was supplied to the leaves that indicated the highest evaporation of moisture from the leaves [3]. The increasing in FIR dryer's temperature may increase the radiation energy that emitted to the leaves and the leaves could absorb more heat. The absorption of this energy resulting in the water vapor pressure inside the leaves thus increases the amount of moisture content evaporated [12]. For leaves that was dried at 40°C and 50°C, both have low moisture content evaporated by the dryer. To get more moisture content evaporated, both drying time of this temperature need to be longer than 6 hour.

Table 1: Moisture content

T (°C)	Initial Weight	Final weight	Moisture content
40	73.10 g	29.99 g	**58.97 %**
50	73.93 g	22.83 g	**69.12 %**
60	72.71 g	15.73 g	**78.37 %**

B. Effect of drying temperature on color of leaves

(a) 40°C **(b) 50°C** **(c) 60°C**

Figure 3: Color of leaves at different drying temperature

Based on Figure 3, it show that there are three different degree of color change based on their drying temperature. This is happen because of chlorophyll degradation during drying process that make the greenness of fresh *A.Malaccensis* leaves decreased [13]. It became darker (more brownish) as the temperature increased until 60°C as show in Figure 3 (a). The color of *A.malaccensis* became less green at higher temperature because magnesium molecules are converted to pheophytin and pyropheophytin [14] as shown in Figure 3 (b).

C. Effect of drying temperature on chemical properties A.Malaccensis leaves using Elemental Analyzer

The remaining samples during extraction process is filtered and dried in order to investigate the CHN amount in the samples. From Figure 4, the highest amount of carbon at temperature 60°C and the smallest amount of carbon at 40°C. There are varies amount of carbon because different temperature have different amount of heat supplied to the leaves during drying process [12]. The higher the temperature, the higher the carbon amount. For hydrogen and nitrogen, the amount of should be decreased when temperature increased but result show on the Figure 4 does not seem correct due to some error while drying the remaining samples from the extraction process. Hydrogen amount represent moisture content value.

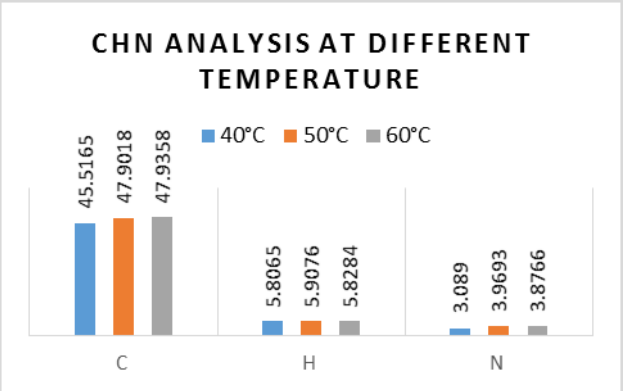
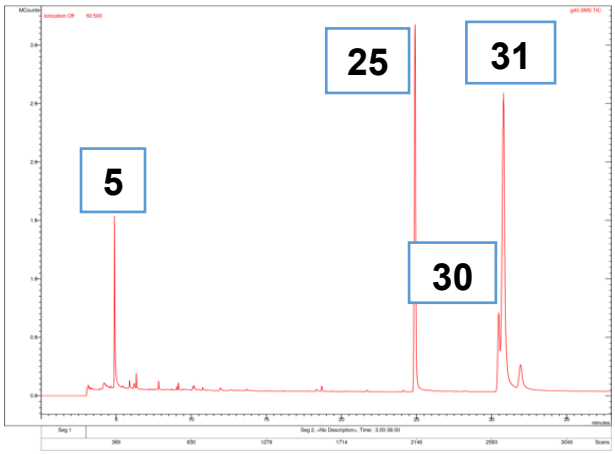


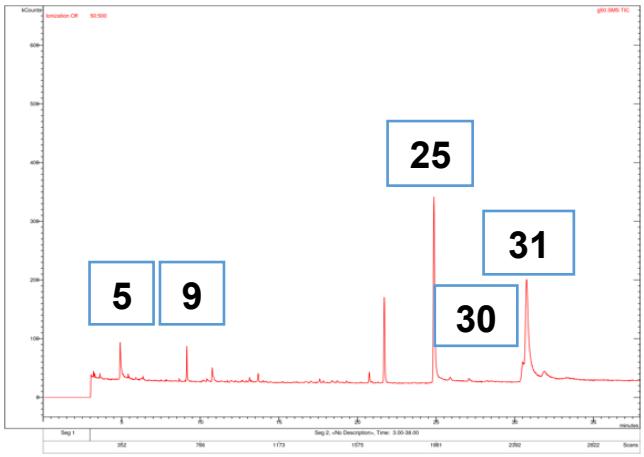
Figure 4: CHN analysis at different temperature

D. Effect of drying temperature on chemical properties A.Malaccensis levaes using GC-MS

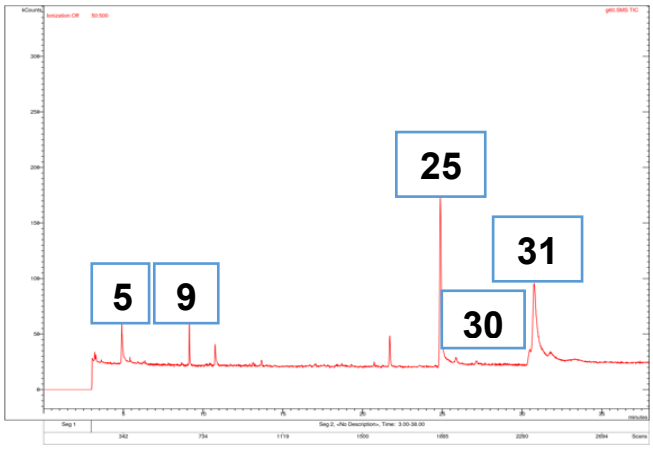
Examinations of three samples of oils with different temperature showed some variations in GC profile and its chemical compound. The variations of GC profiles are shown in Figure 5 (a), Figure 5 (b) and Figure 5 (c) below:



(a) Chromatogram graph at 40°C



(b) Chromatogram graph at 50°C



(c) Chromatogram graph at 60°C

(c) Figure 5: Chromatogram graph

There are 48 compounds were identified from sample 40°C, 35 compounds from sample 50°C and 33 compounds from sample 60°C. For the extraction of *A.Malaccensis* leaves, there are alkaloids, tannins, saponins, flavonoids, terpenoids and phenolic compound [15] were found in GCMS analysis. For example, Mequinol act as phenolic compound and á-Pinene as the terpenoids were found in all samples. In this research, not all compound in sample 60°C were found in sample 40°C and vice versa. This is because there are several factor that cause in different amount and type of chemical compound found in each sample such as different drying temperature of *A.Malaccensis* and duration of extraction. Compound found at drying temperature 60°C is a very little compared at drying temperature 40°C. This due to the damaging of the cell morphology during drying process with the addition of the soaking process too [16]. Table 2 below show the compound that found in the samples:

Table 2: Compounds of *A.Malaccensis* Oil

No	Chemical Compound	40°C	50°C	60°C
1	cis-á-Terpineol	/	/	/
2	à-Phellandrene	/	/	/
3	Ecgonine	/	/	/
4	Citronellyl isobutyrate	/	/	**X**
5	á-Pinene	/	/	/
6	1H-Imidazole, 2-propyl	/	/	/
7	Mequinol	/	/	/
8	ç-Elemene	/	**X**	/
9	6-Octen-1-ol, 3,7-dimethyl-, propanoate	/	/	/
10	Piperidine, 1-methyl	/	**X**	/
11	7-Octen-1-ol, 3,7-dimethyl	/	/	/
12	Estragole	/	/	/
13	Aristolene	/	/	/
14	Limonene oxide, cis	/	**X**	/
15	Naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methyle)	/	/	/
16	Humulane-1,6-dien-3-ol	/	**X**	**X**
17	2-Propanone, 1-(4-methoxyphenyl)	/	/	/
18	1H-Cycloprop[e]azulene, decahydro-1,1,7-trimethyl-4-methylene	/	**X**	**X**
19	5,10-Pentadecadiyn-1-ol	/	**X**	**X**
20	à-Cubebene	/	**X**	**X**
21	1,3-Cyclohexadiene, 5-(1,5-dimethyl-4-hexenyl)-2-methyl	/	/	/
22	1,6-Cyclodecadiene, 1-methyl-5-methylene-8-(1-methylethyl)	/	/	/
23	Naphthalene, decahydro-4a-methyl-1-methylene-7-(1-methylethen)	/	/	**X**
24	Anisaldehyde dimethyl acetal	/	/	/
25	3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)	/	/	/
26	Camphene	/	**X**	/
27	1-Penten-3-one, 1,5-diphenyl	/	/	/
28	Verrucarol	/	/	/
29	3-Carene	/	/	/
30	Squalene	/	/	**X**
31	à-Farnesene	/	/	/
32	Limonene oxide, cis	/	**X**	**X**
33	Ferrocene	/	/	**X**
34	Camphor	/	**X**	**X**
35	(gurjunene) Azulene, 1,2,3,3a,4,5,6,7-octahydro-1,4-dimethyl-7	/	**X**	**X**
36	Naphthalene, 1,6-dimethyl-4-(1-methylethyl)	/	/	/
37	Aromadendrene	/	**X**	**X**
38	1-Eicosene	/	/	**X**
39	ç-Gurjunenepoxide-(1)	/	**X**	**X**
40	6-Octenal, 3,7-dimethyl	/	/	/
41	Phenanthrene, 2-nitro	/	**X**	**X**
42	Estragole	/	/	/
43	Valencene	/	**X**	**X**
44	Anisaldehyde dimethyl acetal	/	**X**	**X**
45	à-Caryophyllene	/	**X**	/
46	6-Octenal, 3,7-dimethyl	/	/	/
47	Azulene, 1,2,3,5,6,7,8,8a-octahydro-1,4-dimethyl-7-(1-methyle	/	/	/
48	á-Panasinsene	/	/	**X**
49	(-)-Spathulenol	**X**	/	/
50	(+)-Epi-bicyclosesquiphellandrene	**X**	/	**X**
51	á-Vatirenene	**X**	/	/
52	Spathulenol	**X**	/	/
53	Caryophyllene	**X**	**X**	/
54	6-Octenal, 3,7-dimethyl	**X**	**X**	/
Total Compound		48	35	33

IV. CONCLUSION

In conclusion, this research has achieved both the objectives targeted at the start of this study where the effect of drying temperature give effect on the chemical and physical properties of *A.Malaccensis* using Far Infrared Radiation (FIR) dryer and the active compound extracted from hydrodistillation method. Based on result, there is a lot of terpenoid found in this 3 sample but it has different amount at every temperature such as at temperature 40°C, 50°C and 60°C, about 48, 35, 33 active compounds were found using GC-MS and the highest amount of carbon were detected at temperature 60°C while using Elemental Analyzer. Therefore, it can be concluded that drying temperature of 40°C give the highest amount of active compound. For recommendation, longer the drying time of the sample at temperature 40°C and 50°C for better result in moisture content evaporated. The soaking time for the sample need to be consider before the hydrodistillation process because it may give effect in the extraction of the oil.

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