EFFECT OF SOAKING ON GAHARU ESSENTIAL OIL YIELD USING MICROWAVE ASSISTED EXTRACTION

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***Abstract*—The extraction of oil can be done using hydrodistillation, steam distillation and supercritical fluid extraction (SFE). However, for this experiment hydrodistillation method is chosen because it is the simplest method but required long time to extract the oil. The objective of this study is to identify the effect of soaking towards the essential oil yield using microwave assisted extraction. The essential oil then will be analyse using gas chromatography-mass spectrometer (GC-MS). The gaharu will be soaked with distilled water at 2,5 and 7 days and will be extracted using MAE. The power of the microwave is constant at 540 W and the temperature is 94.5^{0}C. The time taken for the experiment to run is 12 hours. A total of 3 major compounds which are benzaldehyde, cyclopentane acetic acid and ethanone were identified from the four sample of gaharu residue. The soaking process for 7 days shows the highest amount of component compared to other soaking for 2 and 5 days. This result shows, the cell has expanded and finally damage, thus releasing the content of the soaking water.**

***Keywords*— Hydrodistillation, gaharu, MAE, soaking process, GC-MS**

I. INTRODUCTION

Agarwood, the resinous heartwood from the Aquilaria species (*Thymelaeaaeae*) also called, eaglewood, gaharu, jinko, aloes wood in different cultures [1]. It is called gaharu in Malaysia and Indonesia because in Malay language the gaharu referred to heavy fragrant wood. However, current practice use gaharu as the generic term to refer to both the tree and its resin, similar to term agarwood [2]. Gaharu can be divided into several grades in the market such as super grade A, grade A, B, C and D. The higher quality of the gaharu wood can be recognized by its colour. The dark colour of the wood and the strong aroma released upon burning its chip quality incense. However, there is very little information on the quality of different grades on how the essential oils can be produced [3].

Pretreatment process in the extraction of agarwood oil is a crucial step. Previous study shows several methods to pretreat plant sample before extraction such as soaking in water, chemical treatment, sonification and microwave treatment [4]. Soaking pretreatment is significant in this experiment because it can improve the chemical components extracted and also the yield of essential oils. The chemical components such 3-phenyl-2-butanone, α-guaiene, β-agarofuran can be found if the extracted of soaked gaharu is more than 7 days [5]. Not just that, soaking solvents play a big role in reducing the strength of cell walls and breaking the oil glands [6]. Hence, breaking of the wall is an essential step for the essential oil extraction and prove that soaking activity speed up the process [7]. Next, the soaking process will leach out some acidic component make the solvent now in acidic. This shows that the cell burst, and leech out acidic component make the solvent more acidic [8]. Moreover, another study also discussed the optimize soaking process for different type of *Aquilaria sp* but the agarwood chips does not finely grounded. So, in this work, agarwood chips directly used in the process after it were chipped into smaller pieces [9].

Extraction of oil can be done using hydroditillation, steam distillation, supercritical fluid extraction and many other methods [10]. In Malaysia, usually the method used is hydrodistillation and supercritical fluid extraction (SFE). SFE method used carbon dioxide which similar to hexane in order to extract the oil from the gaharu [11]. Hydrodistillation method in other hand is chosen because the setup is the simplest. For this method, the factors need to be considered were the water present in the vessel must always be enough to last throughout the distillation process or otherwise the plant material, which is gaharu may overheat. For this method, water is use to boil and the essential oil is carried by the steam to condenser to be cooled until it become liquid formed.

The extraction by microwave as assisted is an alternative method compared to ultrasonic. The microwave interacts with the molecule and ions in the material resulting drive physical, chemical and biological reactions [12]. The advantages of using microwave are the purity is very high, increase the reaction rate, the use of the solvent can be reduced and short processing time [13].

One of the application of the gaharu essential oil is to make medicine. The medicine used to treat fever and migraine. The component that can be found in the gaharu is 4-hydroxyacetanilide also known as acetaminophen [14].

II. METHODOLOGY

A. Materials

The raw material which is *Aquilaria Malaccensis* was obtained at Kuala Krai, Kelantan. The solvent used were distilled water supplied from Chemistry Laboratory in Faculty of Chemical Engineering, UiTM Shah Alam.

B. Preparation of Aquilaria Malaccensis from wood to chips

The gaharu wood was cut into small pieces before be grinded to reduce the size particle to smaller size. The crusher (SCP Automation (M) SDN BHD), (model: SLM-20PI/D) were used to grind into size 0.5 – 0.8 cm. Then, the gaharu chips need to be dried to remove the moisture content before stored into zip plastic bag.

C. Preparation of soaking the Aquilaria Malaccensis

350 g of gaharu were soaked into three containers with distilled water. The soaked gaharu need to be pretreated into 2, 5 and 7 days.

D. Microwave Assisted Extraction (MAE)

Microwave assisted extraction was carried out in a microwave digestion system (Microwave-Sharp R-202). In a typical run, the 2-day pretreated gaharu with 350 ml of distilled water (1:10) sample to solvent ratio were introduced into a 5000 ml reaction vessel (Modified Teflon Material) [15]. After that the vessel was placed in the microwave to be heated at power of 540 W. The time taken for each extraction was 12 hours. Then, repeated the experiments with the 5 and 7 days pretreated gaharu.

E. Characterization of the oil, hydrosol and residue of the gaharu

The characterization technique was focused on the composition of the gaharu sample such the oil, hydrosol and residue. Gas chromatography-mass spectrometer was used to identify the element composition for oil, hydrosol and residue. The analytical condition for this experiment are as follows

Table 1: Analytical conditions for GCMS analysis [16]

Program	Conditions
Oven Program, Carrier gas	80 ^{0}C for 2 min, then 10 ^{0}C/min to 250 ^{0}C for 10 min, Helium
Gas flow	2 ml/min
Split ratio	1:50
Injection Volume	1µl
Mode	Splitless
Interface temperature	250 ^{0}C
Electron impact (emission current)	70eV
Scan range	32 to 500 amu

III. RESULTS AND DISCUSSION

A. Effect of Soaking on Component of Gaharu

Three different days of soaking were used in this experiment which is 2,5 and 7 days. Gas Chromatography Mass Spectrometer (GC-MS) was used to determine the component of gaharu residue before and after soaking.

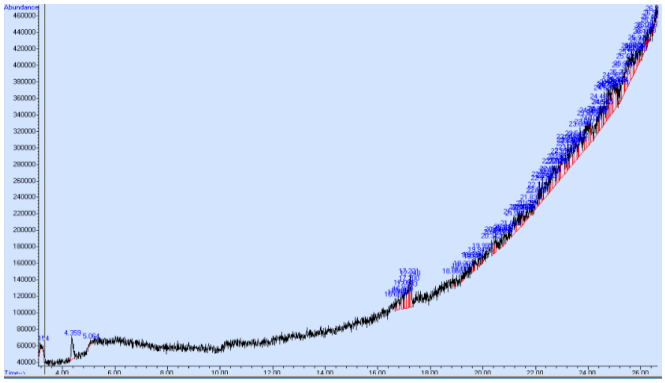

Figure 1: Unpretreated gaharu

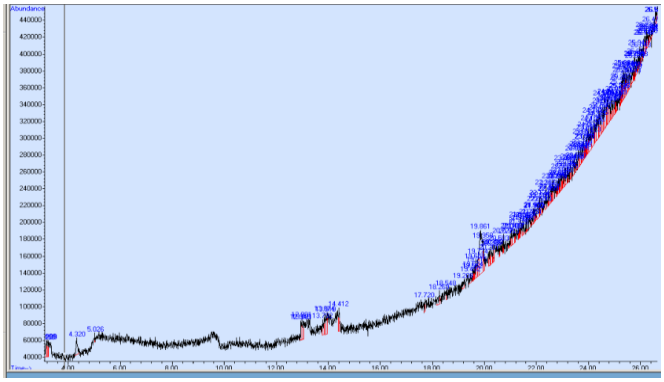

Figure 2: Soaking pre-treatment for 2 days

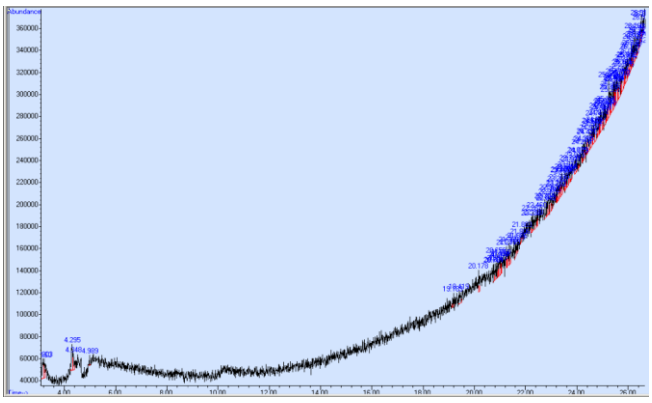

Figure 3: Soaking pre-treatment for 5 days

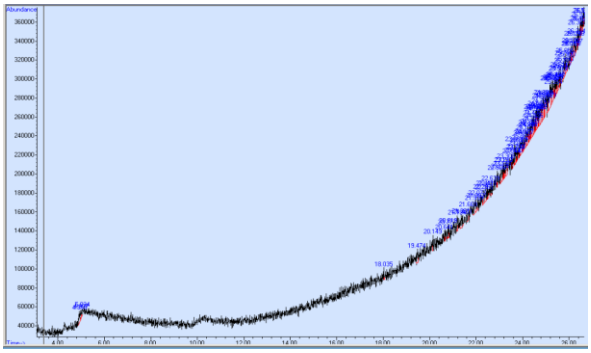

Figure 4: Soaking pre-treatment for 7 days

Figure 1 until Figure 4 shows the GC-MS image of the component of gaharu soaking by using distilled water as solvent. The data was extracted and compiled in a table below.

Table 2: Major Compounds found in Gaharu Residue

Time, min	Compounds	Unpre-treated gaharu	Soaking 2 days	Soaking 5 days	Soaking 7 days
5 to 6	Benzaldehyde	No	Yes	Yes	Yes
19 to 22	Cyclopentane Acetic Acid	Less	More	More	More
21 to 24	Ethanone	Less	More	More	More

Table 2 shows the major compounds that can be found in gaharu residue. Compounds such benzaldehyde, cyclopentane acetic acid and ethenone were the compounds that largely be found in the residue. At retention time 5 to 6 min, the compounds that can be found in the soaking pre-treatment was benzaldehyde. From the result, unpretreated gaharu shows no sign of benzaldehyde compound while the number increase from 4,8 and 10 for soaking process for 2,5 and 7 days.

Next, cyclopentane acetic acid which can be found at retention time between 19 to 22 min. the compounds also show a increase in result when soaking process up to 7 days with 48 times compare to unpretreated gaharu which is only 6 times. Lastly, the major compounds that can be found in gaharu residue was ethenone. These compounds were found between 21 to 24 min of retention time. This ketone family were found at least 3 times during unpretreated gaharu while soaking process for 7 days the compounds can be found up to 76 times.

From the above result, we can conclude that the unpretreated gaharu shows nothing or a small number of major compounds. Different from soaking process, the major component likely to show an increasing in number with the increase of days of soaking. This is because, during soaking process, the cell has expanded and finally damage, thus releasing the content to the soaking water. Water enters cell wall through diffusion and increase the turgor pressure. Soaking water become more acidic through time and corrodes the cell wall.

IV. CONCLUSION

In conclusion, by using GC-MS we have identified different compositions of chemical componenets in agarwood residue from gaharu *A.Malaccensis.* The compounds commonly identified in all four samples which are benzaldehyde, cyclopentaneacetic acid and ethenone. Further studies are needed to refine the results which later can be used to assist detection and authentication as well as its scientific-based grading.

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