# FTIR Analysis of Acetaminophen on Dried *Aquilaria malaccencis* Leaves

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***Abstract*— *Aquilaria malaccencis* leaves is a traditional herbal that has been long used for medicinal remedies. Its various health benefits have also been the main focus on many researches. Acetaminophen is a synthetic drug often used to treat fever and other illness and this compound can be found in *Aquilaria malaccencis* leaves. Currently, there are still lack of studies conducted on the presence of acetaminophen in *A. malaccencis* leaves. Meanwhile, vacuum far-infrared drying (VFIR) has been found to increase drying time and product quality in preservation of food and agricultural products. There are still little studies conducted on the effect of VFIR on *Aquilaria malaccencis* leaves. A study to determine the presence of acetaminophen was conducted by drying the leaves using VFIR dryer at temperature of 30°C, 40°C, 50°C, 60°C and 70°C. The dried leaves were extracted using hydrodistillation method. The presence of acetaminophen in the extracted sample is determined by using Fourier-transform Infrared (FTIR) Spectroscopy. FTIR has been widely used for characterisation of a compounds due to its relatively simple and it allows investigation of functional groups, bonding types, and molecular conformations; hence, providing molecular-level information. A peak from the spectral data was identified at 1640 cm-1 region for all three of the dried extracted sample. An extraction of fresh leaves of *A. malaccencis* leaves were carried out and analysed with FTIR in order to compare it with the extraction of dried *A. malaccencis* leaves. Two additional peaks can be observed and identified at 1498 cm-1 and 1542 cm-1 region. The infrared bands that can be identified from the spectral data belonging in the spectrum of acetaminophen are carbonyl vibration, NH bending and C-NH bending. This shows that there was presence of acetaminophen in *Aquilaria malaccencis* leaves.**

***Keywords*— *Aquilaria malaccencis, Acetaminophen, vacuum far-infrared dryer.***

## I. INTRODUCTION

*Aquilaria malaccencis*, also known as agarwood, is a heavy and fragrant resinous wood formed in Aquilaria tree. This species originates from South and Southeast Asian countries and mainly found in Bangladesh, India, Indonesia, Laos, Malaysia, Myanmar, the Philippines, Singapore, and Thailand [1]. Aquilaria has wide range of applications and often used as incense, perfumery, medicine, religious ceremony, and as ornamentals [2]. Meanwhile, traditional Arab medicine has been using agarwood essential oil for aromatherapy [3]. Its numerous health benefits as a traditional herb have also led to various studies on its medicinal benefits. Studies on Aquilaria leaves have shown that it has various health properties such anti-oxidant, anti-microbial, anti-inflammatory activity and hepatoprotective [4].

Various studies have been conducted on the bioactive compounds in Aquilaria trees, which is important in discovering therapeutic agent in plant. Alkaloids, tannins, flavonoids and phenolic compounds are considered as the most significant phytochemical compounds of plants. The bioactive compounds of plants carry a significant potential in application in human healthcare [5]. Often time, this herb has been widely used to treat pain, relieve fever, rheumatism, and asthma[6].

Fever is known as pyretic and it can be treated by consuming antipyretic drug. Acetaminophen is the most prescribed drug used as antipyretic and analgesic to treat pain and relieve fever [7]. Despite a decline in the use of medicinal plants in the middle of 20th century due to the rise of synthetic drugs, there is a change in trend in people's favour. Medicinal plants are gaining favour as research are focusing on natural products and these plants have long been used are a source of antipyretic agents to treat fever. Due to this matter, there is an urge for more search on potential antipyretic agents from herbal materials [8]. Despite that, there are still lack of studies on the presence of Acetaminophen in *Aquilaria* leaves.

In food preservation, the dehydration technique is probably one of the oldest method practiced by mankind. More rapid drying techniques and methods have been created to decrease the substantial amount of energy needed in the drying process [9]. A drying method of food preservation is usually used to extract the components from *Aquilaria* leaves. Far-infrared radiation (FIR) has gained popularity as a thermal energy source during the past decade for drying of many food and agricultural materials [10]. Previous studies showed that vacuum far-infrared (VFIR) drying does not only reduce drying time but also energy consumption and yield a better dried product quality[9]. Vacuum drying has also been known to quicken the drying process without diminishing the dried product quality. Addition to that, vacuum drying also help prevent oxidation from occurring when air is present [11], [12]. However, there are little studies conducted on the effect of VFIR on *Aquilaria malaccencis* leaves.

Fourier-transform infrared (FTIR) spectroscopy has risen to be one of the major tools for a wide range of applications including analysis of small molecules or molecular complexes to the analysis of cells or tissues [13]. Presently, there are only few works are available reporting the IR spectra of acetaminophen. FTIR is gaining wide interest amongst scientists due to its relatively simple, reproducible, non-destructive to the material, and only require small amounts of material with a minimum sample preparation. Direct information about the chemical composition can be provided as the spectral bands in vibrational spectra are not only molecule specific, but also relatively narrow, easy to resolve, and sensitive to molecular structure, conformation, and environment [14]. The purpose of this research is to evaluate the drying effects of VFIR on *Aquilaria malaccencis* leaves and to investigate the presence of Acetaminophen in the leaves.

## II. METHODOLOGY

### A. Materials

Fresh, undamaged, and matured *Aquilaria malaccencis* leaves

were obtained from a farm located in Jalan Kebun, Shah Alam for the experiment. The leaves were then rinsed with tap water and wiped dried with clean tissues to remove the impurities. The cleaned leaves were kept in a plastic container with a good lid seal and stored in a refrigerator of 4°C prior to start of the experiment.

### B. Experimental Procedures

#### 1) Drying of A. malaccencis using VFIR

Five experimental runs were selected to investigate the effect of vacuum far-infrared dryer drying on the *Aquilaria malaccencis* leaves. A VFIR was used to dry the sample at 5 different temperatures which are at 30°C, 40°C, 50°C, 60°C, and 70°C. The pressure set for this experiment is 0.5 bar. Lower pressure level were selected to ensure faster drying of the sample as evaporation speed of the water is higher at lower pressure [15]. A handful amount of cleaned leaves was taken for each run, placed on the tray inside of VFIR and then dried for 120 minutes to ensure the leaves were well-dried in a fixed residence time.

The leaves sample weighed using weighing balance prior to drying and after drying for 120 minutes to collect data for moisture content removal of the leaves after drying. The moisture content was determined using the following equation:

$$MC = \frac{W - W_1}{W_1} \quad (1)$$

$$MC_{dry\ basis} = \frac{W_{initial} - W_{dried}}{W_{dried}} \quad (2)$$

$$MC_{wet\ basis} = \frac{W_{dried} - W_{initial}}{W_{initial}} \quad (3)$$

#### 2) Extraction of dried A. malaccencis leaves

For this experiment, 3 sets of experiments were conducted for 5g, 10g, and 15g of *Aquilaria malaccencis* leaves that were to be extracted. For 5g of dried sample, 5 sets of temperature were run which were at 30°C, 40°C, 50°C, 60°C and 70°C. Meanwhile, 3 sets of temperature were run for both 10g and 15g which are 40°C, 50°C and 60°C.

The dried samples of *Aquilaria malaccencis* leaves from the temperature of 40°C, 50°C, and 60°C were weighed accurately to different amount of 5g, 10g, and 15g. The samples were extracted using hydrodistillation method and it was performed for 5 hours until the extraction completed.

For the hydrodistillation method, 3 sets of extraction were carried out using samples of 5g, 10g, and 15g. Each set consists of weighed sample from 3 different temperatures which are 40°C, 50°C, and 60°C. Prior to the hydrodistillation, the sample were immersed in 2000 ml of water from a 5000 ml of round bottom flask to avoid charring of the leaves samples. Then, the flask was put into heating mantle and heated up to 150°C for the hydrodistillation process for 5 hours. After the extraction was completed, the extracted samples were collected using vials and stored in a refrigerator at temperature of 4°C.

#### 3) Characterisation of extracted samples using FTIR

The extracted samples were then analysed using Fourier-transform infrared (FTIR) spectroscopy. Perkin Elmer Spectrum One FT-IR model was used for the characterization where the extracted sample was placed on the sample holder. The sample was scanned by the computer and the spectral data obtained for each extracted sample were collected to be analysed.

## III. RESULTS AND DISCUSSION

### A. The effect of VFIR drying on the moisture content of Aquilaria malaccencis leaves

Vacuum far-infrared drying process caused some weight loss of the leaves, showed by the difference of weight of the leaves before and after drying. The loss of weight was attributed to the loss of moisture inside the leaves after it underwent drying process. The main purpose of drying process is to remove the moisture content in order to preserve the ingredients inside the leaves and thus, retain its quality [16].

**Table 1: Effect of VFIR on *Aquilaria malaccencis* for 5g of extraction sample**

| Run | Tempe-rature | Weight of leaves (g) | | % Difference | Moisture content | |
|---|---|---|---|---|---|---|
| | | Before | After | | Dry basis | Wet basis |
| 1 | 30°C | 15.1465 | 13.9110 | 8.07 | 0.0878 | 0.0807 |
| 2 | 40°C | 13.3185 | 9.2283 | 30.71 | 0.4432 | 0.3071 |
| 3 | 50°C | 16.8547 | 6.4223 | 61.93 | 1.6269 | 0.6193 |
| 4 | 60°C | 14.0937 | 5.6200 | 60.12 | 1.5077 | 0.6012 |
| 5 | 70°C | 13.4603 | 5.1833 | 61.54 | 1.6001 | 0.6154 |

**Table 2: Effect of VFIR on *Aquilaria malaccencis* for 10g of extraction sample**

| Run | Tempe-rature | Weight of leaves (g) | | % Difference | Moisture content | |
|---|---|---|---|---|---|---|
| | | Before | After | | Dry basis | Wet basis |
| 1 | 40°C | 31.8433 | 20.2604 | 36.37 | 0.5717 | 0.3637 |
| 2 | 50°C | 29.0262 | 11.0717 | 61.86 | 1.6217 | 0.6186 |
| 3 | 60°C | 30.4882 | 10.9986 | 63.93 | 1.7720 | 0.6393 |

**Table 3: Effect of VFIR on *Aquilaria malaccencis* for 15g of extraction sample**

| Run | Tempe-rature | Weight of leaves (g) | | % Difference | Moisture content | |
|---|---|---|---|---|---|---|
| | | Before | After | | Dry basis | Wet basis |
| 1 | 40°C | 49.7302 | 36.4287 | 26.75 | 0.3651 | 0.3675 |
| 2 | 50°C | 46.3732 | 18.8439 | 59.36 | 1.4609 | 0.5936 |
| 3 | 60°C | 47.6510 | 17.9161 | 62.40 | 1.7720 | 0.6240 |

#### 1) Difference in weight loss after drying process

As shown in Table 1 for batches of 5g extraction sample, the highest percentage of weight loss is run 3 and run 5 using temperature of 50°C and 70°C respectively, with 61.93% and 61.54% loss respectively. Supposedly, the leaves at run 5 should present the highest percentage of weight loss compared to run 3 due to greater moisture removal from the leaves at higher temperature.

During this experiment, the difference in initial weight of leaves sample might have affected the result for dry basis moisture content. The differences in sizes and shapes, including maturity of the leaves sample might also affected the result obtained. Another factor that might have affected the results obtained are the thickness of the leaves. Swasdisevi et al., 2009 reported from their research that increase in thickness of dried product does increases drying time. The reason for this was because the moisture had to travel more before being removed from the surface of dried product. In this case, the leaves samples with varies thickness need more than 120 minutes to sufficiently become dry.

For batches of 10g of extraction sample from Table 2, the highest percentage of weight loss is run 3 using temperature of 60°C with 63.93% loss. Similarly, for batches of 15g of extraction sample as shown in Table 3, the highest percentage of weight loss is run 3 using temperature of 60°C with 62.40% loss. At higher temperature, the moisture removal from the leaves during drying process is greater, hence, explained the increasing trend of weight

difference from lower temperature to higher temperature [16].

*2) Wet basis moisture content of the leaves*

Wet basis moisture content is defined by the percentage equivalent of the ratio of the weight of water to the total weight of the material. It is often used to define the water content of agricultural material and food products. Based on the Table 1, Table 2, and Table 3, an increasing trend can be observed as the temperature used for drying process increases. For batches of 5g extraction sample, run 3 and run 6 have the highest wet basis moisture content, at 0.619329 and 0.615403 respectively using temperature of 50°C and 70°C respectively.

A similar increasing trend in wet basis moisture content can be observed for both batches of 10g of extraction sample and 15g of extraction sample. With the increase in drying temperature, the rate of moisture reduction increases, and increases the wet basis moisture content [11].

*3) Dry basis moisture content of the leaves*

Dry basis moisture content is defined by the percentage equivalent of the ratio of the weight of water to the weight of dry matter. It is commonly used for describing moisture changes during drying. For batches of 5g extraction sample, the highest dry basis moisture content is run 3 with value of 1.626941 using temperature of 50°C.

Theoretically, dry basis moisture content changes linearly with the weight loss or gain when a sample loses or gains moisture. During drying process, moisture is loss and the amount of moisture removed from sample is higher with increasing temperature [10]. Supposedly, with increasing temperature, dry basis moisture content would decreases; which means the moisture content would showcase a decreasing trend as temperature reaches 70°C [17].

This consideration may also apply for batches of 5g and 10g of extraction sample since it showed similar trend of increasing dry basis moisture content.

## *B. Characterisation of Acetaminophen using Fourier-transform Infrared Spectroscopy (FTIR)*

Figure 4.1 shows the FTIR spectra of 5g of extracted *Aquilaria malaccencis* leaves after being dried using VFIR at temperature of 40°C, 50°C, and 60°C. From the spectral data in the figure, there was a strong peak in 3200-3400 $cm^{-1}$ region which showed the infrared bands of water. Since water has a broad band, it has a very strong absorption in the 3700-3100 $cm^{-1}$ region.

Another peak from the spectral data is at 1640 $cm^{-1}$ region which can be observed for all three of the extracted sample. For 5g of extracted sample dried at temperature of 40°C, 50°C, and 60°C, the peak is observed at 1638 $cm^{-1}$, 1640 $cm^{-1}$, and 1643 $cm^{-1}$ respectively. This infrared band is estimated and can be identified in the spectrum of acetaminophen which is the carbonyl vibration. Carbonyl vibration infrared band can be identified at 1637.2 $cm^{-1}$ in the spectrum of acetaminophen. Other major bands identified in the spectrum of acetaminophen were the aromatic ring vibration at 1517.5 $cm^{-1}$ and the OH deformation-C-O stretch at 1243.2 $cm^{-1}$. Other bands that can also be identified in the spectrum of this compound is H3C-C=O stretching at 1378.4 $cm^{-1}$, NH bending at 1447.9 $cm^{-1}$ and C-NH vibration at 1552.2 $cm^{-1}$[18].

A similar data was obtained from the analysis of 10g of extracted sample and 15g of extracted sample. The spectral data from both of extracted samples as seen from Figure 4.8 and 4.9 showed similar peaks which can be estimated and identified as carbonyl vibration in the spectrum of acetaminophen at range of $1630^{-1}650$ $cm^{-1}$. Both data also showed strong absorption at 3260-3320 $cm^{-1}$, which are the infrared bands of water.

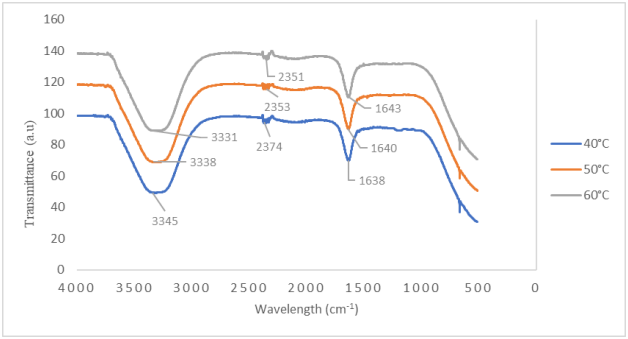


**Figure 4.1: FTIR spectra of 5g of dried extracted *Aquilaria malaccencis* leaves**

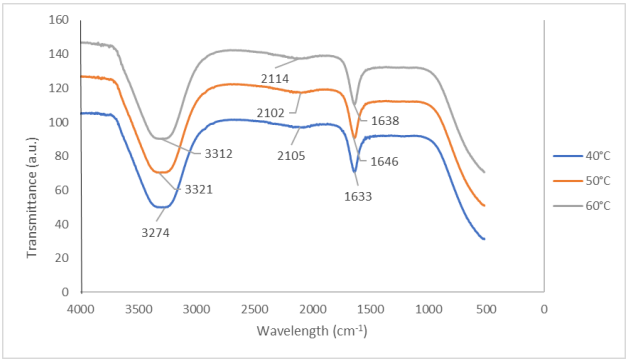


**Figure 4.2: FTIR spectra of 10g of dried extracted *Aquilaria malaccencis* leaves**

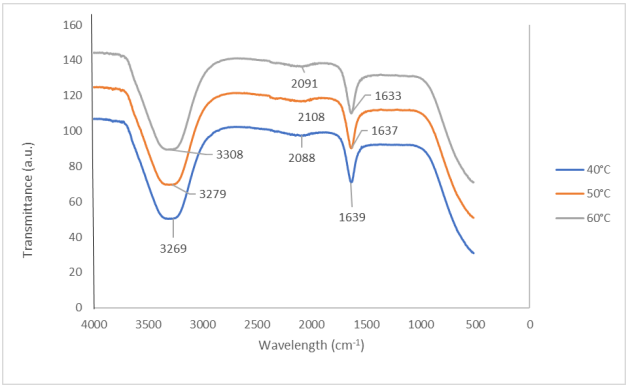


**Figure 4.3: FTIR spectra of 15g of dried extracted *Aquilaria malaccencis* leaves**

From the Figure 4.1, 4.2, and 4.3, only infrared band of carbonyl vibration can be observed and identified in the spectrum of acetaminophen. The lack of other major bands presence from this spectrum might due to lack of sufficient essential oil extracted from the dried leaves samples. Most of the extracted and contain only little of essential oil. The reason for this matter might due to the fact that the capacity used for the hydrodistillation extraction process was too large which was at 5L capacity instead of 2L capacity. Since the capacity for the extraction was too large, the amount of water used for hydrodistillation is larger compared to the amount of dried leaves samples that were to be extracted. Therefore, there were only little amount of oil produced and may not be sufficient enough for characterisation of Acetaminophen using FTIR.

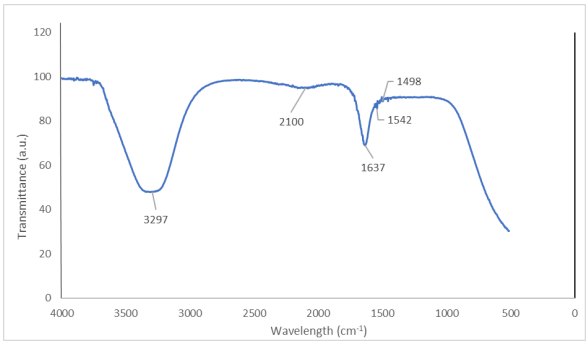


**Figure 4.4: FTIR spectra of extracted *Aquilaria malaccencis* fresh leaves**

An extraction of fresh leaves of *Aquilaria malaccencis* were carried out and analysed with FTIR in order to compare it with the extraction of dried A. malaccencis leaves. The spectral data obtained was plotted as shown in Figure 4.4. Similar infrared bands from the extraction of dried leaves sample were observed from the result obtained which are the strong absorption of water infrared band at 3297 $cm^{-1}$, and the carbonyl vibration at 1637 $cm^{-1}$ region. However, there were another two peaks than can be observed and identified which are at 1498 $cm^{-1}$ and 1542 $cm^{-1}$ region. Both peak can be estimated as NH bending (1498 $cm^{-1}$) and C-NH vibration (1542 $cm^{-1}$). According to Ramos et al, 1998, these two infrared bands are found in the spectrum of acetaminophen with NH bending at 1447.9 $cm^{-1}$ and C-NH vibration at 1552.2 $cm^{-1}$ region.

Based on the data obtained, it can be concluded that by drying the A. malaccencis leaves prior to extraction, it may have degenerated the phytochemical compound in the leaves. As a result of it, there were less infrared bands in the spectrum of acetaminophen that can be identified from the extracts of dried leaves compared to the extracts of fresh leaves. The infrared bands identified from the extracts of fresh leaves can be found in the spectrum of acetaminophen which indicates the presence of acetaminophen in the *Aquilaria malaccencis* leaves.

Apart from that, there was a weak peak found at 2100-2350 cm-1 region on all four of the extracted samples. This infrared band is estimated and can be identified as -C≡C- which is in functional group of alkyne. The characteristic absorption of alkyne group is in the range of 2100-2260 cm-1.

## IV. Conclusion

From the data tabulated in Table 1, 2, and 3 from Results and Discussion section, it is clear that *Aquilaria malaccencis* leaves dried at higher temperature had greater moisture loss. The leaves sample that are dried at 60°C had the highest weight loss difference, which indicates the most removal of moisture from the leaves. At higher temperature, the dried products should also have the highest value of wet basis moisture content and the lowest value of dry basis moisture content as rate of moisture reduction of the leaves increases.

Extraction method is needed in order to analyse phytochemical content from the leaves samples. The extracted samples then can be analysed using FTIR since it allows investigation of functional groups, bonding types, and provide molecular-level information. Based on the spectral data obtained from using FTIR, presence of acetaminophen in dried extracts of A. malaccencis leaves can be determined. After drying, few infrared bands from the spectrum of acetaminophen can be identified from the spectral data. However, FTIR analysis on extract samples of fresh A. malaccencis leaves presented more identified peaks that shows infrared bands in the spectrum of acetaminophen. The infrared bands that can be identified from the spectral data belonging in the spectrum of acetaminophen are carbonyl vibration, NH bending and C-NH bending. The presence of these infrared bands indicates the presence of acetaminophen in *Aquilaria malaccencis* leaves.

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