EXTRACTION PERFORMANCE STUDY OF GAHARU USING MICROWAVE EXTRACTION METHOD

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Thesis submitted in partial fulfilment of the requirements for the award of the degree of Bachelor of Chemical Engineering

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this project and in my opinion, this project is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

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STUDENT'S DECLARATION

I hereby declare that the work in this project is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

SIGNATURE :

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Dedicated to my late father, my late mother and my family.

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ABSTRACT

Nowadays, as technology develops, more process and equipments is found in order to extract gaharu especially for its essential oil. In producing high yield of essential oil with less time consuming, this study is important so that the extracted product can be used in multiple ways as in varies industry and are suitable for pharmaceutical and herbs industry. This study is limited to two parameters which are days of gaharu soaking and the microwave power. The gaharu grade C used will be grind and soak in the distilled water before extract and the oil yield will be analyze using Fourier transform infrared spectroscopy (FTIR). From this study, it have been proven that microwave extraction is suitable in extracting gaharu with acceptably low time comparing to other extraction equipment but the yield is too little and as gaharu itself contains low resin in it, the oil extracted were mostly visible at the wall of equipments. As the power of microwave increases, the yield obtained increases too due to the loosening of cell wall of gaharu that increase the yield essential oil. The days of soaking has also been proven to be directly proportional to the yield of oil extracted, considering the power of microwave set and other condition.From the analysis done by FTIR, several compounds have been found such as sulfoxide, alkanes, alkyl halides, alcohol and imines. Although the component of gaharu itself can't be ascertain, the existence of alcohol in some of the mixture shows that there might be possibilities of the chemical compound of gaharu such as jinkoh-eremol, agarospirol and jinkohol that were OH group miscible in the distilled water analyze.

ABSTRAK

Pada masa kini, dengan berkembangnya teknologi, pelbagai proses dan peralatan telah dijumpai untuk mengestrak gaharu terutamanya untuk mendapatkan minyak pati. Dalam menghasilkan hasil yang tinggi minyak pati dengan mengambil masa yang kurang, kajian ini adalah penting supaya produk yang diekstrak boleh digunakan dalam pelbagai industri dan sesuai untuk industri farmaseutikal dan herba. Kajian ini terhad kepada dua parameter yang merupakan hari gaharu direndam dalam air suling dan kuasa gelombang mikro yang digunakan. Gaharu gred C yang digunakan akan dikisar dan direndam di dalam air suling sebelum diekstrak dan hasil minyak akan dianalisis menggunakan inframerah spektroskopi jelmaan Fourier (FTIR). Dari kajian ini, ia telah membuktikan bahawa pengekstrakan gelombang mikro yang sesuai dalam mengeluarkan gaharu dengan masa rendah berbanding dengan lain-lain peralatan pengekstrakan tetapi menghasilkan minyak yang terlalu sedikit dan oleh kerana gaharu itu sendiri mengandungi resin yang rendah di dalamnya, minyak yang diekstrak kebanyakannya hanya dapat dilihat di dinding peralatanApabila kuasa mikro yang ditetapkan meningkat, hasil yang diperolehi meningkat. Ini disebabkan oleh dinding sel gaharu yang telah lemah menyebabkan minyak pati yang terhasil meningkat. Hari gaharu direndam juga telah terbukti berkadar terus dengan hasil minyak diekstrakDaripada analisis yang dilakukan oleh FTIR, beberapa sebatian telah dijumpai seperti sulfoxide, alkana, alkil halida, alkohol dan imines. Walaupun komponen gaharu itu sendiri tidak boleh ditentukan, kewujudan alkohol di beberapa campuran menunjukkan bahawa mungkin ada sebatian kimia gaharu seperti jinkoh-eremol, agarospirol dan jinkohol dalam kumpulan OH yang larut dalam air yang telah dianalisis.

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LIST OF SYMBOLS

m	Gradient
^{o}C	Degree
g	Gram
atm	Atmosphere
W	Watt

LIST OF ABBREVIATIONS

FTIR	Transform Infrared Spectroscopy
MAE	Microwave Assisted Extraction
SCFE	Supercritical extraction method

CHAPTER 1

INTRODUCTION

1.1 PROBLEM STATEMENT

Gaharu is a resin product which is produced by particular product and has a certain high commercial value but is threatened to extinction due to uncontrolled exploitation (Tarjuman M., 2011). It has at least 300 year in the Middle East, China and Japan and is used by Traditional Chinese, Unanai, Ayuravedic, and Tibetan physicians. They are used for the purpose of medical, which is the remedy for nervous system disorders such as neurosis, and exhaustion (Robert *et al*, 2004). There are many grades of agarwood, and the highest quality of the wood is extremely expensive. As a matter of fact, the first-grade wood is one of the most expensive natural products in the world, with prices of up to $13,000 per pound (Eden Botanicals, 2007).

Nowadays, though the Agarwood is also used for medical purpose, but as technology develops, more process and equipments is found in order to extract it, especially for its essential oil. However, throughout the year, there were still problems in extracting gaharu. As the goal is to obtain high yield of gaharu essential oil with reasonably low time in producing it, many equipment have been build and develop. Compared to other conventional method used such as ultrasonic assisted steam distillation by Ahmad Junaidy Jaapar 2008, the condition of high ratio of gaharu-water (1:20) and long extraction hour (9h) will be solved in this experiment by using microwave extraction.

By using microwave extractor to extract the gaharu, the extraction time is shorten and the reaction will consume less solvent (Jain T. *et al*, 2009). As no residue generated and no/small quantity water or solvent is use in the extractor, cleaner feature gives an advantages as there will be no risk that there will be impurities in the reaction.

1.3 RESEARCH OBJECTIVE

The objectives of this study are to study the performance of microwave extraction in extracting gaharu essential oil and the effects of microwave power, and days of gaharu soaked in distilled water to the yield extracted.

1.3 SCOPE OF THE STUDY

Extraction time, gaharu sizes, days of soaking, and the condition of the microwave extractor such as power, temperature and pressure are the parameters that can be manipulated in studying the performance of microwave extraction for extracting essential oil. However, this study is limited to two parameters which are days of gaharu soaking and the microwave power.

1.4 RATIONALE AND SIGNIFICANCE

In producing high yield of essential oil with less time consuming, this study is important so that the extracted product can be used in multiple ways as in varies industry and productions such as perfume, incense and in medicine. In the same time the study can suggest better extraction method, by which means the extraction time is short and that it produce reasonably amount of essential oil. The product yielded is also a fine chemical produced, as it is in high quality, which is suitable for pharmaceutical industry, as well as herbs industry. Increasing efficiency will increase the rate of profit gain from the manufacturing and processing of the essential oil. In particular, the study will help Malaysian Herbs Industry in producing more quality product.

CHAPTER 2

LITERATURE REVIEW

2.1 GAHARU

2.1.1 Gaharu

Gaharu or Agarwood is the resin-impregnated wood that is derived from the Aquilaria species of the family of Thymalaeacea. There are many names given for this resinous wood, including agar, agarwood, aloeswood, kalamabak and eaglewood (Angela.B et al. 2003). Other names such Eaglewood, Aloes Wood and Agalocha is also used (Broad, 1995) but it is commonly referred to as Gaharu in Malaysia. Agarwood is a large evergreen tree growing over 15-30 m tall and 1.5-2.5 m in diameter, and has white flowers (Chakrabarty et al., 1994). It is well known for its unique fragrant and highly valuable non-timber products in Asian tropical forest. The odor of Agarwood is complex and pleasing with few or no similar natural analogues.

The formation of agarwood occurs in the trunk and roots of trees are due to the infection by a parasite ascomycetous mould, Phialophora parasitica, a dematiaceous (dark-walled) fungus. The trees occasionally begin to produce an aromatic resin in response to this attack. As the fungus grows the resin dramatically increases the mass and density of the affected wood, changing its color from pale beige to dark brown or black within the heartwood (Ng et al., 1997). The oldest of the cultivated trees containing the oleoresin are 12 years of age whereas the resin found in wild harvested trees has been developing for 30 years or more. So the age of the raw material has a lot to do with the richness and complexity of the resin produced therein. However, there are many merits in the oil from the cultivated trees (Ng et al., 1997).

Figure 2.1: Picture of gaharu.

Table 2.1: Common Names of Agarwood

Language	**Names**
English	Agarwood, Aloeswood, Eaglewood
French	Bois d’aigle, Bois d’aloes
Vietnamese	Tram Huong
Chinese	Chen-xiang
Japanese	Jinkoh
Arabic	Oud
Indonesian	Gaharu
Malay	Gaharu
Thai	Kritsana noi

Source: Gaharu Online, n.d.

2.1.2 Distribution by country and habitat

The species of gaharu has a wide distribution, being found in Bangladesh, Bhutan, India, Indonesia, Malaysia, Myanmar, the Philippines, Singapore and Thailand. (Review of significant trade, 2003). Around the tropical region there has been reported that 15 species of Aquilaria has exists in India, Burma, China, Myanmar and Malaysia region. In Malaysia there are 5 species of Aquilaria found which are Aquilaria Hirta, Aquilaria Malaccensis, Aquilaria Rostrata, Aquilaria Microcorpa and Aquilaria Becanana. A significant number of research studies have been conducted on Aquilaria malaccensis (Ng et al., 1997) which is well distributed throughout Peninsular Malaysia except for Kedah and Perlis.

Five species of Aquilaria are recorded for Peninsular Malaysia and all are believed to be able to produce oleoresins (Cheng et al.2001)as shown in Table 2.2;

Table 2.2: gaharu producing species of Aquilaria in Peninsular Malaysia (Chang *et al*,2001)

Species	**Local name for resinous wood**	**Grade**
A.Malaccensis	Gaharu	Medium
A.microcarpa	Garu	
A.hirta	Chandan	Medium
A.rostrata	-	-
A.beccariana	Gaharu, tanduk	-

It has also been said that in peninsular Malaysia, Orang Asli has been the traditional harvester of gaharu and that in east Malaysia, Penans people are the traditional gatherers of gaharu (Hansen, 2000).

2.1.3 Physical properties

Chemical profile for each grade such as grade A, B and C were different. In peninsular Malaysia, the gaharu were mostly made of grade C quality (Chang et al., 2002). Different chemical component in gaharu oil will determine the characteristic or quality of the gaharu. Gaharu can be distinguished by colors, specifically ‘reddish brown’ and ‘greenish brown’ (Fatmawati Adam et al, 2005).

Table 2.3: Physical properties of Agarwood oil (sensibly depend on species of Aquilaria tree, mainly for color, odor, viscosity and specific gravity).

Properties	**Description**
Odor	sweet aromatic scent
Appearance	Lightly yellow to brownish liquid
Specific gravity (@25oC)	0.89 - 1.08 g/cm^3
Refractive index (@20^{o}C)	1.4910 - 1.6090
Optical Rotation (@20^{o}C)	-13.2o until -17.80
Acid value	6.8 -13.2
Ester value	18.3 - 27.1
Solubility	soluble in alcohol

2.1.4 Importance/uses

2.1.4.1 Medicine

Agarwood has been used for medicinal purposes for thousands of years, and continues to be used in Ayurvedic, Tibetan and traditional East Asian medicine (Chakrabarty et al., 1994; Fratkin, 1994). The Sahih Muslim, which dates back to approximately the eighth century, refers to the use of agarwood for the treatment of pleurisy and its use is referenced in the Ayurvedic medicinal text the Susruta Samhita. Agarwood is prescribed in traditional East Asian medicine to promote the flow of qi', to relieve pain, arrest vomiting by warming the stomach, and to relieve asthma (Anon., 1995a). High-grade agarwood powder is prescribed in Chinese medicine (Yaacob, 1999) and is also used in the production of pharmaceutical tinctures (Heuveling van Beek and Phillips, 1999). Burkill, 1966 reported that Malaysians used agarwood mixed with coconut oil as a liniment, and also in a boiled concoction to treat rheumatism and other body pain.

Bull 1930, cited in Chakrabarty et al., 1994 that Agarwood is use as a complex ointment for smallpox and for various abdominal complaints. It is also prescribed for dropsy, as a carminative, a stimulant, for heart palpitations, and as a tonic taken particularly during pregnancy, after childbirth and for diseases of female genital organs (Chakrabarty et al., 1994). By Burkill 1966, and Okugawa et al., 1993, it have been said that gaharu is also believed to have tonic and therapeutic properties which can cure rheumatism, shortness of breath, general pains, diarrhea, asthma and a lot more.

2.1.4.2 Perfume

The use of agarwood for perfumery extends back several thousands of years, and is referenced, for example, in the Old Testament several times using the term 'aloes'. Both agarwood smoke and oil are customarily used as perfume in the Middle East (Chakrabarty et al., 1994). It is also used in Arabic countries as well, where it is in high demand. Unlike many industrial perfumes, the agarwood oil is suitable for hot climates. It is by far the most precious essential oil with prices reaching as much as ten times that of sandalwood oil.

In spite of its unique qualities though, Agarwood is rarely used in European perfumeries because of its cost and good quality synthetic substitutes are yet to be created. Agarwood perfumes are seldom pure agarwood oil, but instead use an alcoholic or non-alcoholic carrier, such as sandalwood oil. The cheapest agarwood perfumes are either synthetic or a blend of oils, each with different qualities and fragrances. Although there are several commercially available synthetic agarwood fragrance compounds, they can produce only low-quality agarwood fragrances, owing to the chemical structure of natural oil (Heuveling van Beek and Phillips, 1999).

Agarwood essences have recently been used as a fragrance in soaps and shampoos (Kadir et al., 1997), cited in Schippmann, 1999. Agarwood is said to have been highly prized by European perfumers in the mid-1990s (cited in Chakrabarty et al., 1994).

(Figure 2.2: Gaharu Technologies, 2011)

2.1.4.3 Incense

Irregular chunks of agarwood usually a few centimeters long and weighing 10-200 g, may be cut or broken into smaller pieces and then burned, usually in a specially made incense burner (Heuveling van Beek and Phillips, 1999). In Middle East, the Egyptians are believed to have used agarwood incense as part of their death rituals more than 3,000 years ago (Person, 2009). It is also used for various purposes in the Middle East, especially during prayers (Yaacob, 1999). In Japan, a revival in the ancient art of Koh doh, the incense ceremony, has revitalized interest in agarwood (Katz, 1996).

The Taiwanese consumers purchase agarwood for the manufacture of incense sticks, which are used in Agarwood perfume, chips and powder and use it in prayers during many traditional festivals and ceremonies to bring safety and good luck (TRAFFIC East Asia-Taipei, in litt. to TRAFFIC International, 2 May 2000) In Malaysia, Muslims burn agarwood splinters or chips to produce incense during special religious occasions, particularly at gatherings, and agarwood incense has been recorded in use there during Ramadan prayers (Chakrabarty et al., 1994). Some Malay tribes fumigate paddy fields with agarwood smoke to appease local spirits (Chakrabarty et al., 1994).

2.1.4.4 Others

Burkill, 1966 reported that grated agarwood has also used in Malaysia for cosmetic purposes. Twine is reported to be made from Aquilaria in Malacca, a province of Malaysia (Chakrabarty et al., 1994). The use of agarwood bark as a writing material has also been documented extensively and agarwood is used for chronicles of important and sacred religious books. The use of gaharu wood as a substitute for paper is also known from the mountaineers of Annam (Vietnam) and from China (Chakrabarty et al., 1994).

Aquilaria bark was reportedly used for this purpose during the nineteenth century (Heuveling van Beek and Phillips, 1999). Some foresters in India have suggested using Aquilaria wood for constructing tea-boxes (Chakrabarty et al., 1994). There are a considerable number of craft shops offering religious ‘agarwood’ sculptures, usually Buddhist figures. Although a proportion of immature agarwood is used in this trade, most statues are not made with agarwood, owing to its soft and flaky properties, which make it unsuitable for carving. Solid pieces of agarwood are highly appreciated as ‘natural art’ in Japan, Korea and Taiwan. Craftsmen carve raw pieces of agarwood into beautiful wooden sculptures. Moreover, agarwood is also turned into beads and bracelets (Person, 2009).

2.2 ESSENTIAL OIL

2.2.1 About essential oil

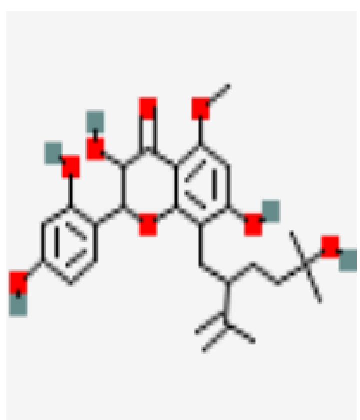
Essential oils, like all organic compounds, are made up of hydrocarbon molecules and can further be classified as terpenes, alcohols, esters, aldehydes, ketones and phenols etc (Nor Azah Mohd Ali, 2002). The terpenes in Gaharu oil can be further divided into monoterpenes and sesquiterpenes. Most monoterpenes in Gaharu oil have a structure consisting 10 carbon atoms and at least one double bond. Terpenes react readily with air in the presence of even the smallest heat source. This is the reason citrus oils degrade quickly unless properly stored. Sesquiterpenes on the other hand consist of 15 carbon atoms and have complex pharmacological actions which include anti-inflammatory and anti-allergy properties. Professor Otto Wallach attributes the fragrance of Gaharu oil mostly to the presence of terpenes and cites the terpenes as having greatly influenced the oil industry. In addition, for oxygenated compounds, they are contains phenols and alcohols such as monoterpene and sesquiterpene alcohol. The phenols found in essential oils normally have a carbon side chain and here we can look at compounds such as thymol, eugenol and carvacrol. These components have great antiseptic, antibacterial and disinfectant qualities and also have greatly stimulating therapeutic properties.

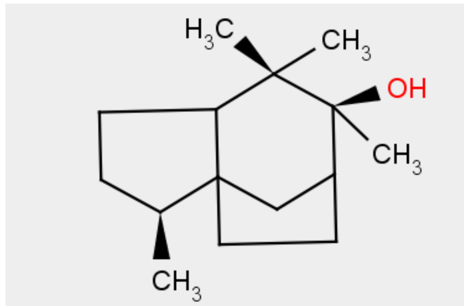
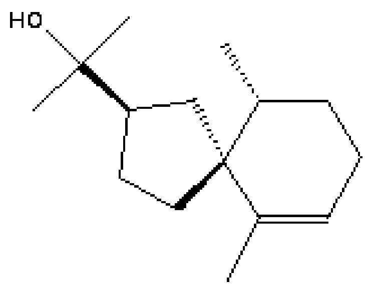
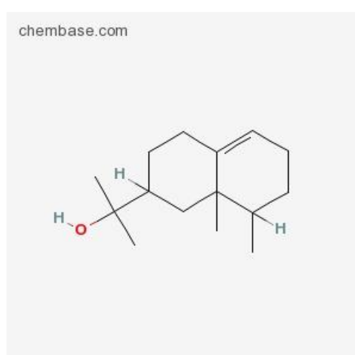
2.2.2 Chemical components

Some of important compounds of gaharu are agarospirol, jinkohol-eremol, jinkohol and kusenol which contribute to the characteristic aroma of gaharu (Nakanishi et al., 1984, Ishihara et al., 1993). Specifically the chemical compounds of interest discovered in Aquilaria Malaccensis Benth are α-agarofuran, 10-epi-γ-eudesmol 6.2%, agarospirol 7.2%, jinkohol 5.2%, jinko-eremol 3.7%, khusenol 3.4%, jinkohol II 5.6%, and oxoagarospirol

3.1% (Yoneda et al, 1984, Nakanishi et al, 1984) but in generally, gaharu oils are mixture of sesquiterpenes, sesquiterpene alcohols, oxygenated compounds, chromone derivatives and resins. Other compounds such as 2-(2-4'-methoxyphenylethyl) chromone also produce a long lasting fragrance upon burning.

Table 2.4: Chemical constituent in gaharu (Pubchem, 2005)

Chemical constituent in gaharu	**IUPAC name/molecular formula**	**Molecular weight(MW)**
Khusenol	2-(2,4-dihydroxyphenyl)-3,7-dihydroxy-8-(5- -elicoid-5-methyl-2-prop-1-en-2-yl-hexyl)-5-methoxy-chroman-4-one 	472.527 g/mol

Jinkohol	C15H26O  (tcm.cmu.edu.tw, *n.d.*)	222.366 g/mol
Agarospirol	2-(6,10-dimethyl-2-spiro[4.5]dec-9- enyl)propan-2-ol  (Caslab.com,*n.d.)*	222.366 g/mol
Jinkoheremol	2-(8,8a-dimethyl-2,3,4,6,7,8-hexahydro-1H-elicoids-e-2-yl)propan-2-ol  (Chembase.com,*n.d.)*	222.366 g/mol

2.3 EXTRACTION

2.3.1 Types

Extraction is a separation process to separate the desired product solute and/or to removed undesired solute compound from the solid where the solid is contacted with a liquid phase. There are many equipment have been develop and build for the purpose of extraction. This section will discuss more on the types of extraction equipment such as microwave assisted extraction (MAE) , steam distillation and supercritical fluid extraction method(SFCE).

2.3.2 Microwave Assisted Extraction (MAE)

Microwave assisted extraction (MAE) is based on the direct application of electromagnetic radiation to a material. It has an ability to absorb electromagnetic energy and to transform it into heat. Unlike conventional heating by infrared energy or thermal conductivity, the increase in temperature occurs simultaneously in the whole volume of solvent. This process is caused by the multiple collisions of the solvent molecules as they realign in the oscillating electromagnetic field, which generates energy in the form of heat. Compared with other extraction methods such as ultrasonic extraction and Soxhlet extraction, MAE are reported to be a higher recovery of the analyte, shorter extraction times and the use of smaller quantities of solvent. In open systems, the extraction occurs at atmospheric pressure and with variable energy input. In closed systems, extraction takes place at controlled pressure, up to 5 atm and a temperature that may exceed the boiling point of the solvent under atmospheric conditions, to increase extraction efficiency.

There are many factors affecting the efficiency of microwave extraction. Solvent used, time of application of microwave, microwave power level, and temperature are the parameters that can be manipulate to obtain optimum condition of the extraction (R.Winny *et al*, 2011)

Below are the figures of microwave and its component;

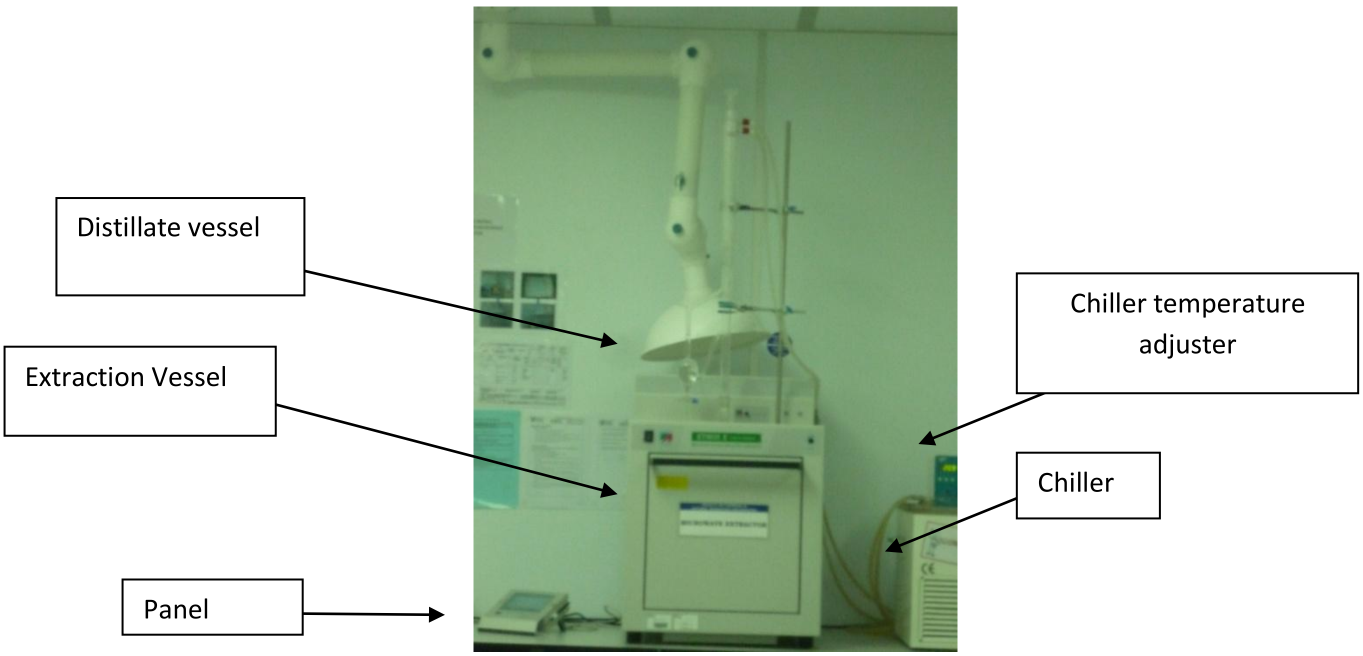


Figure 2.3 : Microwave extraction picture

The chiller must be set 10 oC and waited for reach the temperature before the extraction can be start. This is to ensure the safety of the equipment as well as the person using it. Panel is the place where the parameter and the condition of the experiment is set. There, the heating profile obtained can also be seen. The sample will be put inside the beaker inside the extraction vessel and the oil will goes up to the distillate vessel.

Figure 2.4: Chiller temperature adjuster

Figure 2.5: The distillate vessel.

2.3.3 Steam distillation

This type of distillation is used for temperature sensitive materials like natural aromatic compounds, which is commonly used and a conventional method in extracting oil. The process is simple, that as long as the temperature and pressure is monitored throughout the process, then the steam will remain at its nominal condition. However, as compared to supercritical extraction method, the use of additional filtration or centrifugation is needed to remove the residue.

2.3.4 Supercritical extraction method(SCFE)

It is a new separation technique in chemical engineering that has been develop in the last decade. It is applicable to extract and refine nonvolatile, and thermal sensitive natural material. The end result of this extraction is that we will get a super concentrated and high quality version of essential oil as solvent used, CO2 will returned back to gaseous state leaving no residue remains.

2.4 ANALYSIS OF COMPOUND

2.4.1 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) can provide very useful information about functional group. The technique can be used to analyze organic materials and some of inorganic materials. The FTIR technique is to measure the absorption of various infrared radiations by the target material, to produces an IR spectrum that can be used to identify functional groups and molecular structure in the sample.

The Sample Analysis Process contains below normal instrumental process:

i) The Source: Infrared energy is emitted from a glowing black-body source. This beam passes through an aperture which controls the amount of energy presented to the sample (and, ultimately, to the detector).

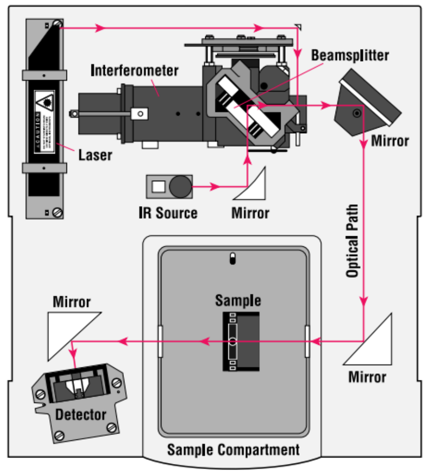
ii) The Interferometer: The beam enters the interferometer where the "spectral encoding" takes place. The resulting interferogram signal then exits the interferometer.

iii) The Sample: The beam enters the sample compartment where it is transmitted through or reflected off of the surface of the sample, depending on the type of analysis being accomplished. This is where specific frequencies of energy, which are uniquely characteristic of the sample, are absorbed.

iv) The Detector: The beam finally passes to the detector for final measurement. The detectors used are specially designed to measure the special interferogram signal.

v) The Computer: The measured signal is digitized and sent to the computer where the Fourier transformation takes place. The final infrared spectrum is then presented to the user for interpretation and any further manipulation.

Below is the simple spectrometer layout;



(Thermonicolt.com,*n.d.)*

Figure 2.6: Picture of FTIR analyzer.

There are several uses of FTIR that will be use in the research such as to identify unknown materials, to determine the quality or consistency of a sample and to determine the amount of components in a mixture.

CHAPTER 3

METHODOLOGY OF RESEARCH

3.1 PROCESS FLOW CHART

Below are the processes of the extraction;

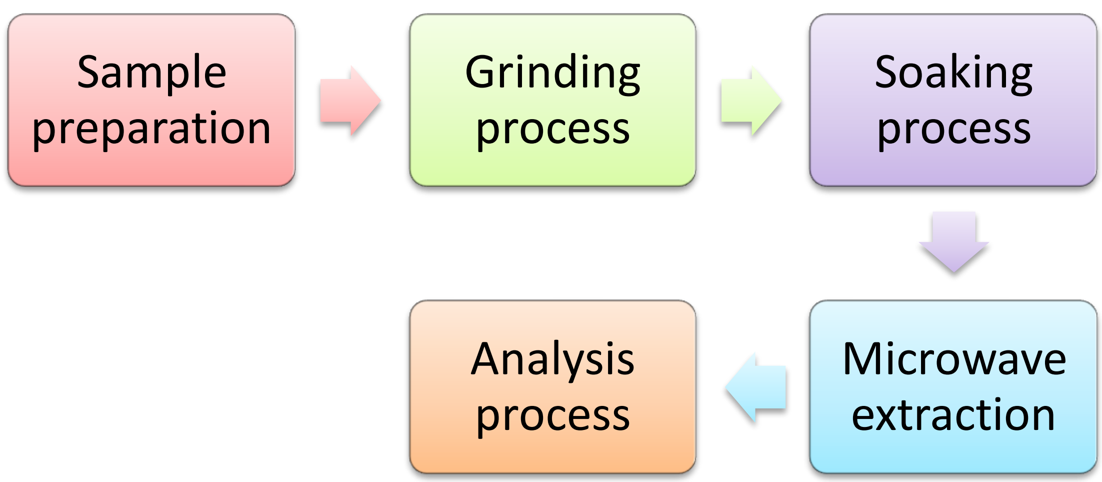


Figure 3.1: Flow process of the research

3.2 SAMPLE PREPARATION

Forest Research Institute Malaysia (FRIM) has made a study that gaharu wood can actually be divided into several grades, which is A, B, and C (Chang et al, 2002). Based on the research regarding gaharu grade using image processing technique, it have been said that the grade of gaharu is determined by its color, which dark gaharu means it is more valuable than the lesser (A.Azma, 2007). For this research, the gaharu that will be used is grade C. This is because; the cost of raw material is lower than those in higher grade. The purpose of using gaharu grade C and not the other grade, which means higher grade, is because in grade A and B, the percentage of resin composition is higher than the percentage wood composition.

Figure3.2: Picture of gaharu sample, grade C

3.3 GRINDING PROCESS

This process is usually run after the drying process is completed. However, since the gaharu obtained from the supplier isn't wet there was no need for drying to be made. The pieces of gaharu wood are ground into sawdust with approximately 1mm in size. This is to maximize the surface area of the gaharu and to ease the extraction process. The condition is supported by the theory that rate of extraction is directly proportional to the contact area surface which means that more particle of gaharu grind will enhance the rate of extraction process. The pieces of gaharu wood are put into the grinder through the hole at the upper part of the grinder.

Figure 3.3 : Picture of grinder

3.4 SOAKING PROCESS

Soaking is a treatment process before preceding the gaharu to the extractor. Gaharu will be soaked with distilled water for 7 days, in order to break down the parenchymatous cells and oil glands. In this experiment, the gaharu time of soaking will be the parameters hence the time will be vary which were 2 days, 5 days, and 7 days of soaking.

3.5 MICROWAVE EXTRACTION

Microwave Assisted Extraction (MAE)

Microwave assisted extraction (MAE) is based on the direct application of electromagnetic radiation to a material. It has an ability to absorb electromagnetic energy and to transform it into heat. Unlike conventional heating by infrared energy or thermal conductivity, the increase in temperature occurs simultaneously in the whole volume of solvent. This process is caused by the multiple collisions of the solvent molecules as they realign in the oscillating electromagnetic field, which generates energy in the form of heat. Compared with other extraction methods such as ultrasonic extraction and Soxhlet extraction, MAE are reported to be a higher recovery of the analyte, shorter extraction times and the use of smaller quantities of solvent. In open systems, the extraction occurs at atmospheric pressure and with variable energy input. In closed systems, extraction takes place at controlled pressure, up to 5 atm and a temperature that may exceed the boiling point of the solvent under atmospheric conditions, to increase extraction efficiency.

There are many factors affecting the efficiency of microwave extraction. Solvent used, time of application of microwave, microwave power level, and temperature are the parameters that can be manipulate to obtain optimum condition of the extraction (R.Winny *etal*,2011).

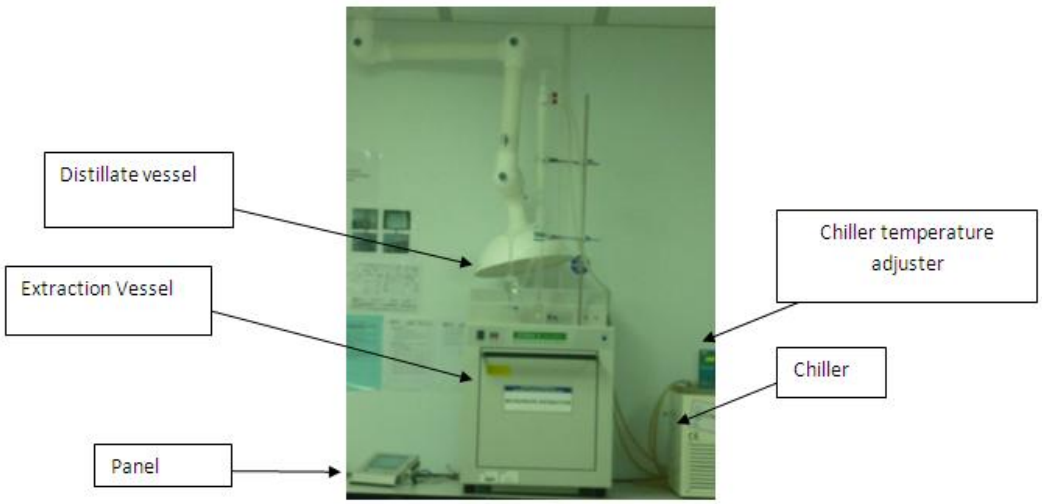


Figure 3.4 : Picture of microwave extraction

In the experiment, there are two parameters involved which are microwave power and the days of soaking. The experiment will be run as Table 3.1 shown below;

Table 3.1 : Condition of experiment for each sample

No of sample	**Weight of gaharu after soaking (g)**	**Gaharu + distilled H2O weight (g)**	**Microwave power (W)**	**Weight of gaharu after extraction (g)**	**Weight of oil extracted (g**	**Oil yield (g)**	**Oil yield (%)**
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							

3.6 ANALYSIS OF PRODUCT

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform spectroscopy is a measurement technique whereby spectra are collected based on measurements of the coherence of a radiative source, using time-domain or space-domain measurements of the electromagnetic radiation or other type of radiation. By using the FTIR, one can characterize the spectrum of a light source in which how much light is emitted at each different wavelength. The most straightforward way to measure a spectrum is to pass the light through a monochromator, an instrument that blocks all of the light *except* the light at a certain wavelength. Then the intensity of this remaining (single-wavelength) light is measured. The measured intensity directly indicates how much light is emitted at that wavelength. By varying the monochromator's wavelength setting, the full spectrum can be measured. This simple scheme in fact describes how *some* spectrometers work.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

In the study, the extraction is done using the Microwave extraction method. The experiment was run for 90 minute for each sample. The gaharu sample that was extracted is added 50mL of distilled water that acts as solvent and the extraction is run at 100 ^{o}C. Two parameter was set which are microwave powers and days of gaharu soaked in distilled water.

The parameter of the experiment is as shown as in Table 4.1 below;

Table 4.1: Parameters and condition of the experiment.

Soaking time	2, 5, 7 days with 1: 7 gaharu-distilled water ratio
Microwave power	300, 500, 700 W

4.2 GAHARU ESSENTIAL OIL YIELD CALCULATION

Based on the experiment done, the oil yields is too little and are visible stuck at the microwave apparatus.

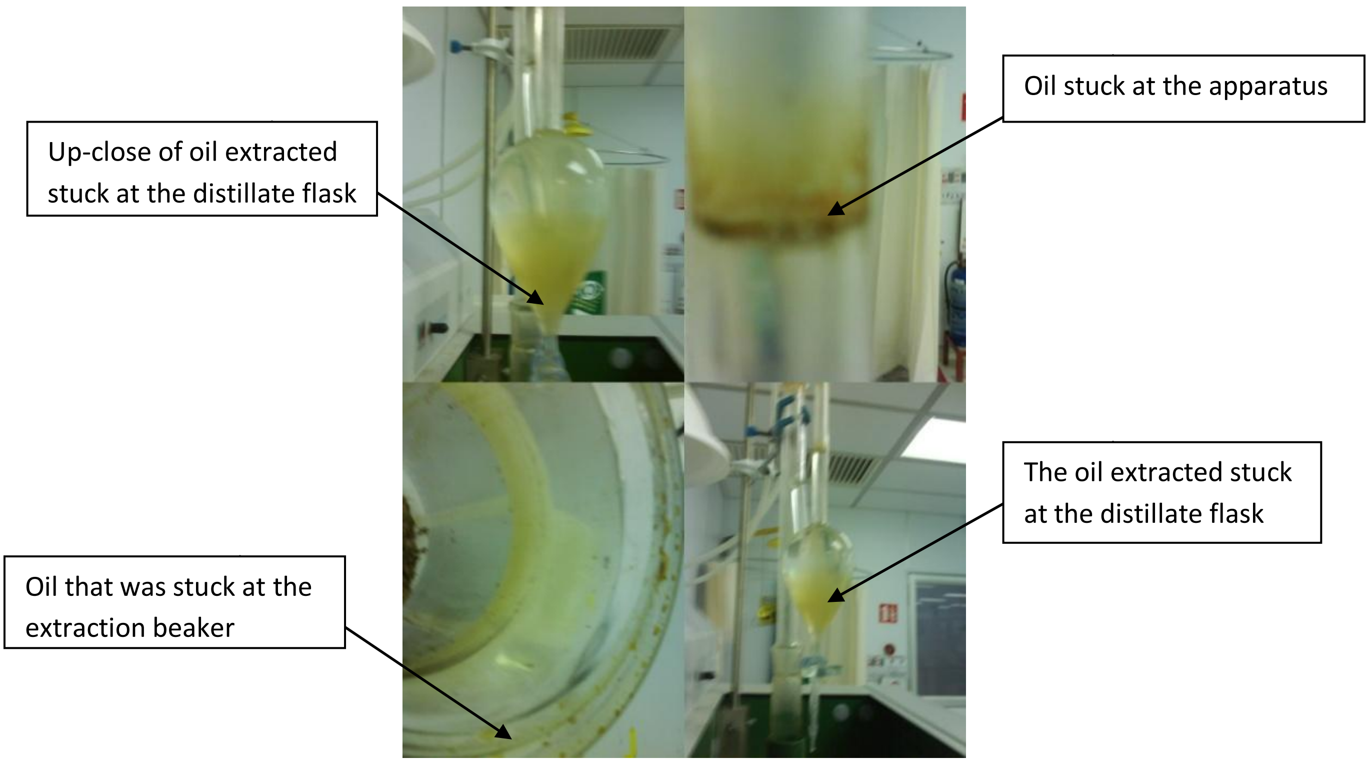


Figure 4.1: The essential oil extracted stuck on the wall of apparatus.

The reason that the yield extracted is too small is that the extraction incomplete recovery of essential oil from raw material and loss of dissolve essential oil in condensate or distilled water. It is observed that all distilled water in distillate have smells of gaharu which proven that some of the essential oil is dissolved in distilled water.

The way to calculate the yield of gaharu essential oil is as below;

$$\text{Yield} = \frac{\text{Amount of essential oil (g)obtained}}{\text{Amount of gaharu oil used (g)}} \times 100\ \%$$

Amount of essential oil (g) obtained = (Weight of gaharu + distilled water) – Weight of gaharu after extraction

Based on the information from MARDI, it is common that the average yield of oil is as in Table 4.2 below. Knowing that gaharu yield will be a lot less, therefore

Table 4.2: Percentage of oil yield recorded for various plants.

Type of oil	Percentage of oil yield(%)
Patchouli oil	0.65
Tea tree	1.3
Lemon grass	0.4
Cinnamon	0.4

(MARDI,2011)

Below are the oil yield calculations of the experiment;

Table 4.3: Yield calculation for the extraction

No of sample	Soaking time (day)	Weight of gaharu after soaking (g)	Gaharu + distilled H2O weight (g)	Micro-wave power (W)	Weight of gaharu after extraction (g)	Weight of oil extracted (g)	Oil yield (g)	Oil yield (%)
1	5	250	298.13	700	183.65	114.48	0.45792	0.004579
2	5	250	296.54	900	192.73	103.81	0.41524	0.004152
3	5	250	296.24	500	203.03	93.21	0.37284	0.003728
4	5	250	294.91	300	210.44	84.47	0.33788	0.003378
5	2	250	300.22	700	164.69	135.53	0.54212	0.005421
6	2	250	301.25	900	155.18	146.07	0.58428	0.005842
7	2	250	297.65	300	274.48	23.17	0.09268	0.000926
8	2	250	297.93	500	211.19	86.74	0.34696	0.003469
9	7	250	304.01	900	183.92	120.09	0.48036	0.004803
10	7	250	299.89	700	238.14	61.75	0.247	0.00247
11	7	250	302.51	500	255.83	46.68	0.18672	0.00186
12	7	250	304.34	300	287.2	17.14	0.06856	0.000685

4.3 ANALYSIS OF ESSENTIAL OIL YIELD CALCULATED

This section will discuss the relationship of parameters chosen to the yield of essential oil extracted. Below are the graph of percentage of oil yield vs. microwave power for varies value of days of the gaharu soak in distilled water. The sample gaharu is soaked for 2, 5 and 7 days with the ratio of 1:7 of gaharu to distilled water and the extraction were done in 90 minutes.

Table 4.4: Oil yield obtain for various values of microwave power and soaking time

Soaking time (days)	**Microwave power (W)**	**Oil yield (%)**
5	300	0.00338
5	500	0.00373
5	700	0.00458
5	900	0.00415
2	300	0.00093
2	500	0.00347
2	700	0.00542
2	900	0.00584
7	300	0.00069
7	500	0.00187
7	700	0.00247
7	900	0.00480

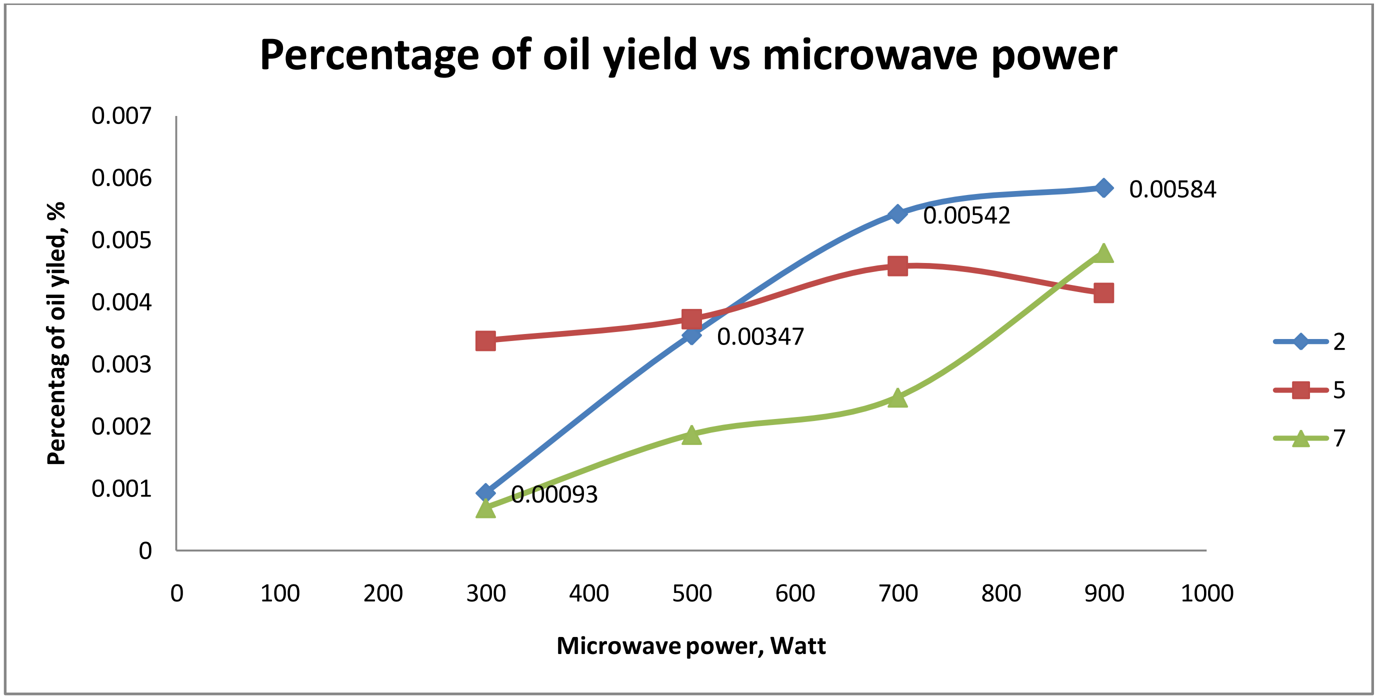


Figure 4.2: The percentage of oil yield vs microwave power for various days of soaking

In general, as the experiment continues, for each set of soaking time the yield obtain is directly proportional to the microwave power. The more power value is set, the higher the percentage of yield obtained. Research made by Mandal *et al*, 2007 shows that in some cases, the soaking of dried plant material in the extracting solvent prior to MAE has resulted in improved yield of oil caffeine by 0.49% and green tea leaves by 1.53%. It have also been said that natural moisture content of the matrix improves the extraction recoveries.

This can be seen from the line of 7 days of soaking whereby the yield obtain is increasing smoothly prior to the given power of the microwave. Comparing the gradient of 2 days of soaking line and 7 days of soaking line, and calculating it at each 500W, 700W and 900W, the result obtained are as Table 4.5 below;

Table 4.5: Gradient (m)of 7 days and 2 days of soaking

Gradient, m = Difference of % of oil yield / Difference of microwave power	2 days of soaking	7 days of soaking
500 W and 300 W	1.270 E-5	5.900 E-6
700 W and 500 W	9.750 E-5	3.000 E-6
900 W and 700 W	2.100 E-6	1.165 E-5

By quantitative calculations, the table above can be interpreted by finding their respective gradient differences, whereby in line 2 days of soaking the difference is both 1.30 and 4.64 and for line 7 days of soaking the difference is both 1.97 and 0.26. The situation can be seen clearly from the graph that the increasing yield obtained for 2 days of soaking is much more rapid than in 7days of soaking. This thus shows the pattern of those lines rising.

Theoretically, microwave radiation loosens the cell wall matrix and leads to severing of the parenchyma cells, thereby the skin tissues are rapidly and extensively opened up by the microwave. (Kratchanova, 2004)This will lead to increased interaction between extracting agent and source material in the extraction process. As a result, permeation of the extracting agent will be increased thus leads to effective increase in the yield of gaharu essential oil. From the graph itself, it can be seen that for each of the soaking day of gaharu in the distilled water, the graph is continuously increase as the power of the microwave increased. For 2days of soaking line, in the beginning of the experiment the yield improved with a sharp rate and then by approaching to an equilibrium state, there was a gradual increase in the yield. However, that doesn't apply to both of 5 and 7 days of soaking.

For the line of 5 days of soaking, at 700 W, the yield obtains are 0.00458 but is declining for 900W with yield of 0.00415. This is in agreement to the studies made by the Mandal, Mohan and Hemalatha on the Review article of microwave assisted extraction which stated that high power with prolonged exposure always involves the risk of thermal degradation. The situation is because rapid rupture of cell wall takes place at higher temperature when kept at higher power therefore the impurities are also leached out into solvent together with the desired analytes. This means that at 900W, for 5 days of soaking the cell have been degraded and are no longer effective for extraction.

4.4 ANALYSIS OF FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY GRAPH

Below are the graphs of the FTIR obtains from the experiment done for the experiment;

Sample 1;

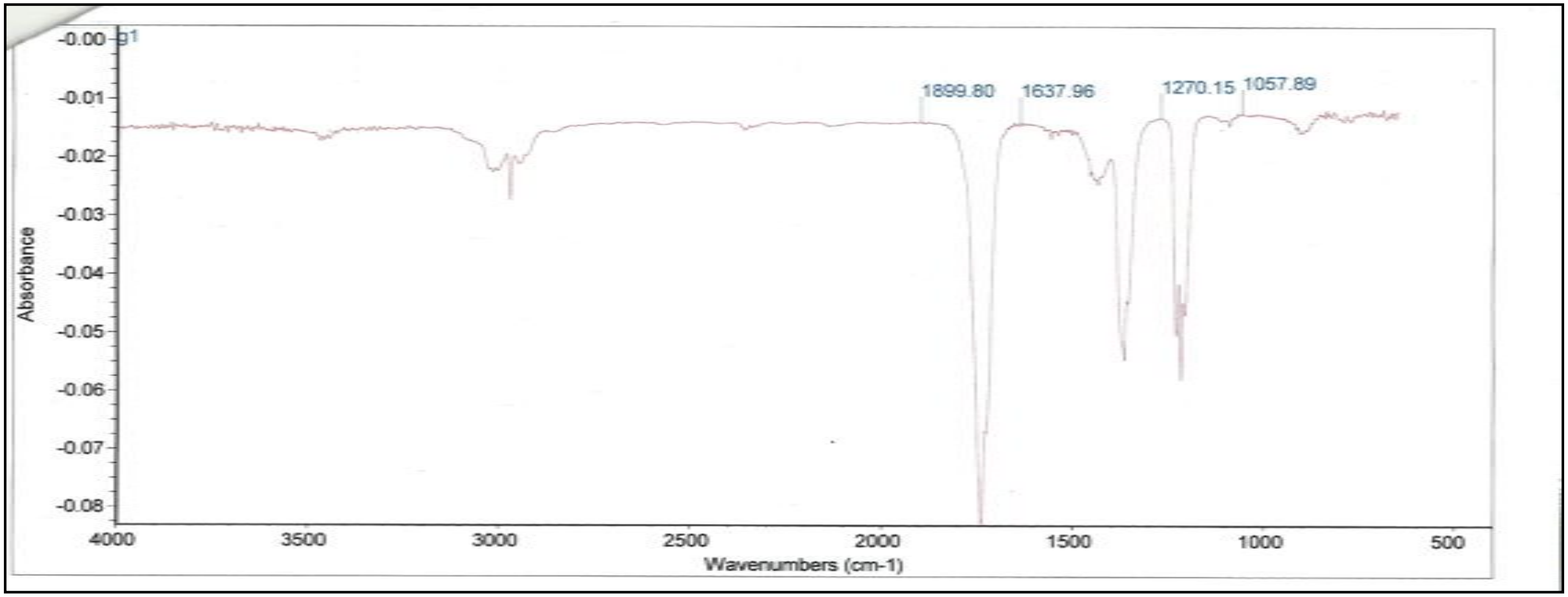


Figure 4.3: Graph of FTIR sample 1

Sample 2;

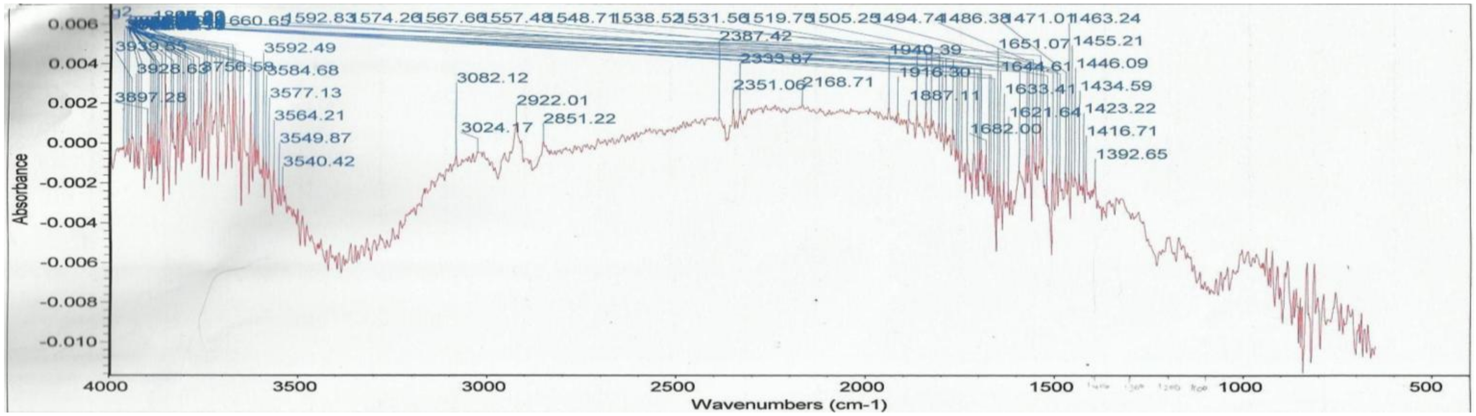


Figure 4.4: Graph of FTIR sample 2

Sample 3;

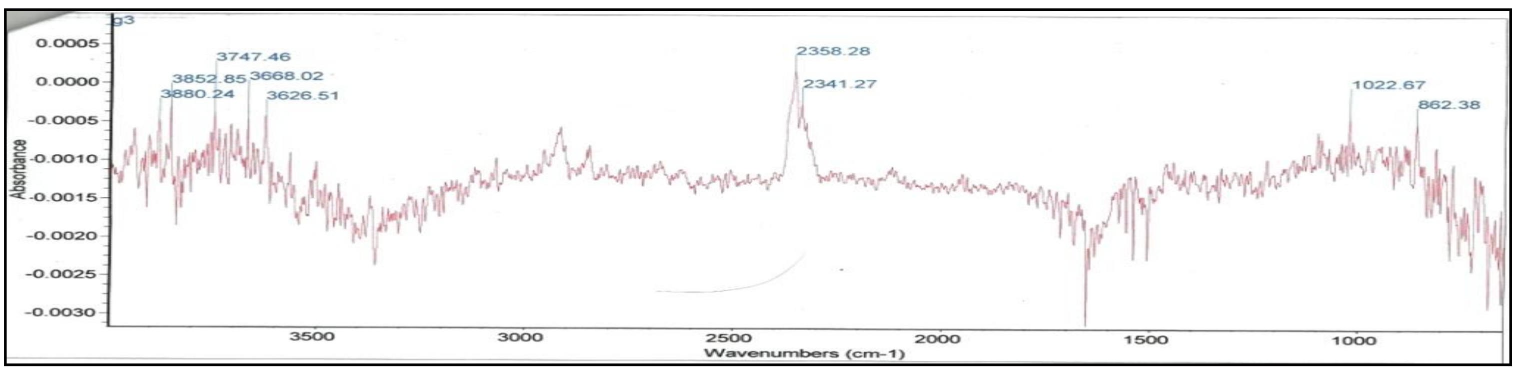


Figure 4.5 Graph of FTIR sample 3

Sample 4;

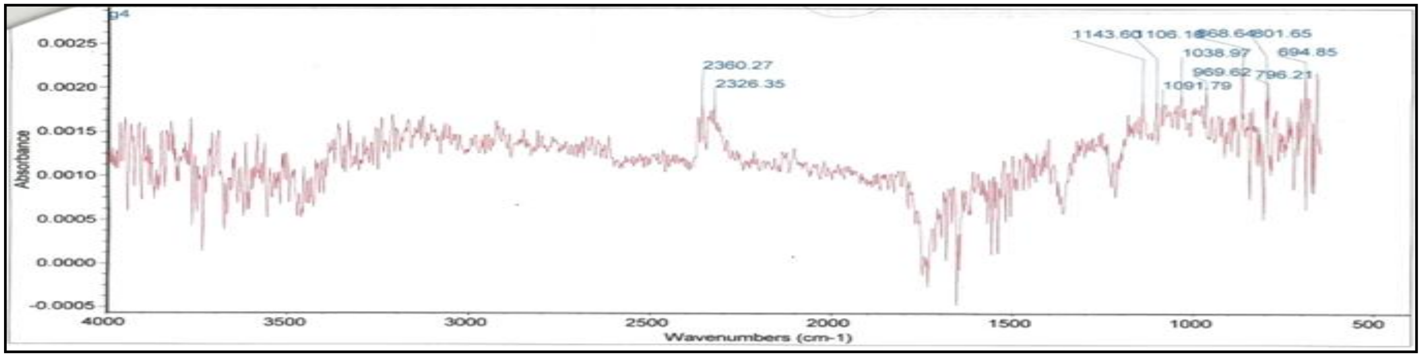


Figure 4. 6 Graph of FTIR sample 4

Sample 5;

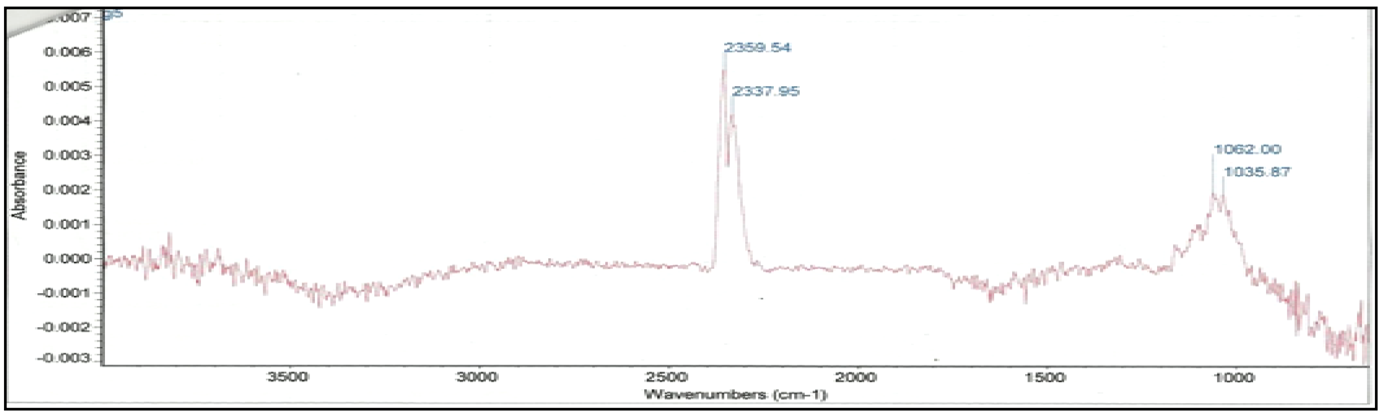


Figure 4. 7 Graph of FTIR sample 5

Sample 6;

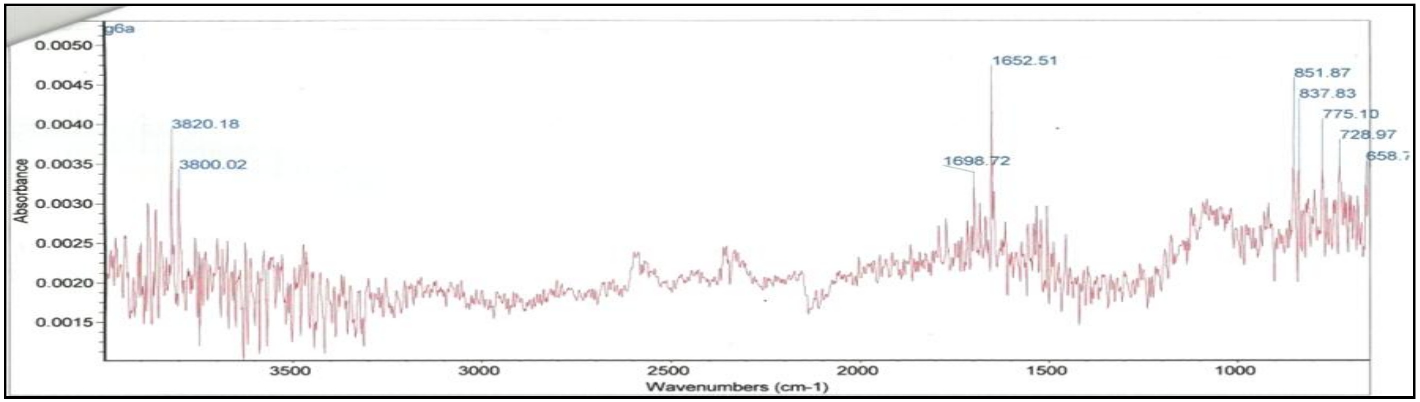


Figure 4.8; Graph of FTIR sample 6

Sample 7;

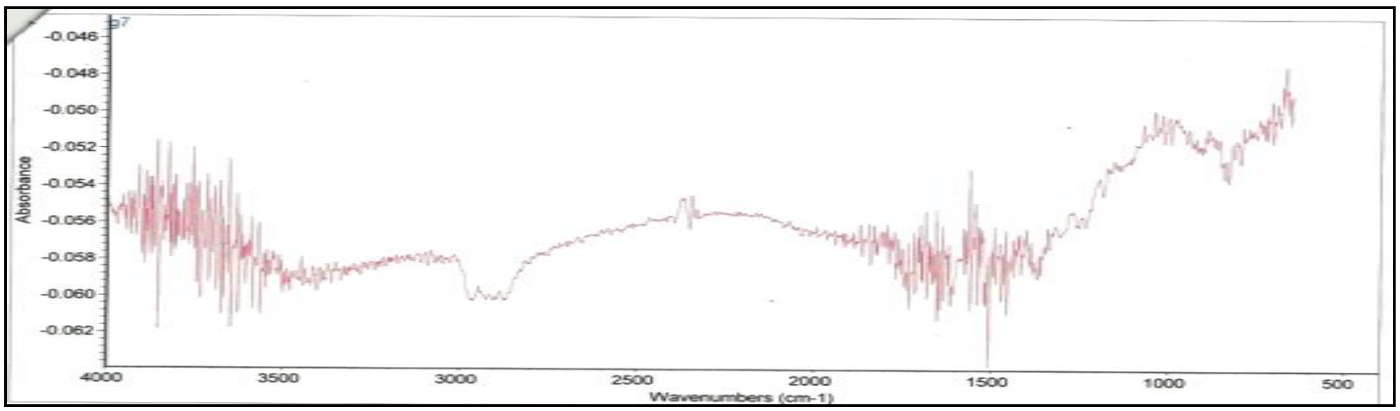


Figure 4. 9 Graph of FTIR sample 7

Sample 8;

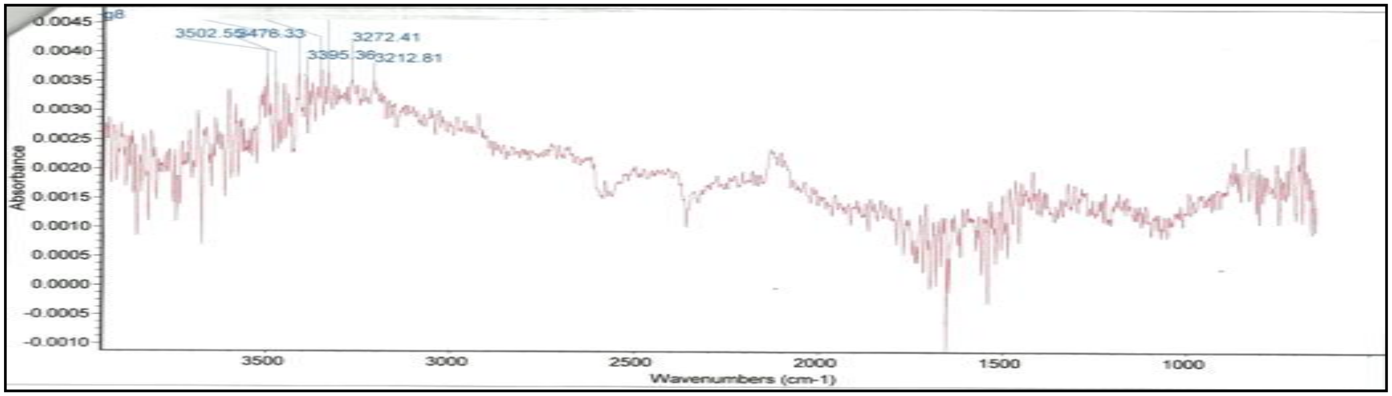


Figure 4. 10 Graph of FTIR sample 8

Sample 9;

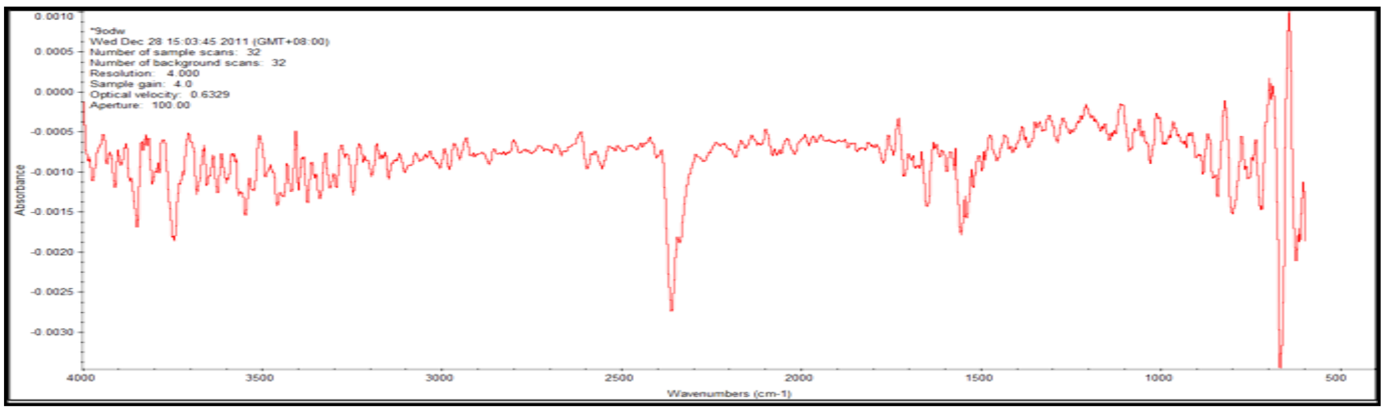


Figure 4. 11 Graph of FTIR sample 9

Sample 10;

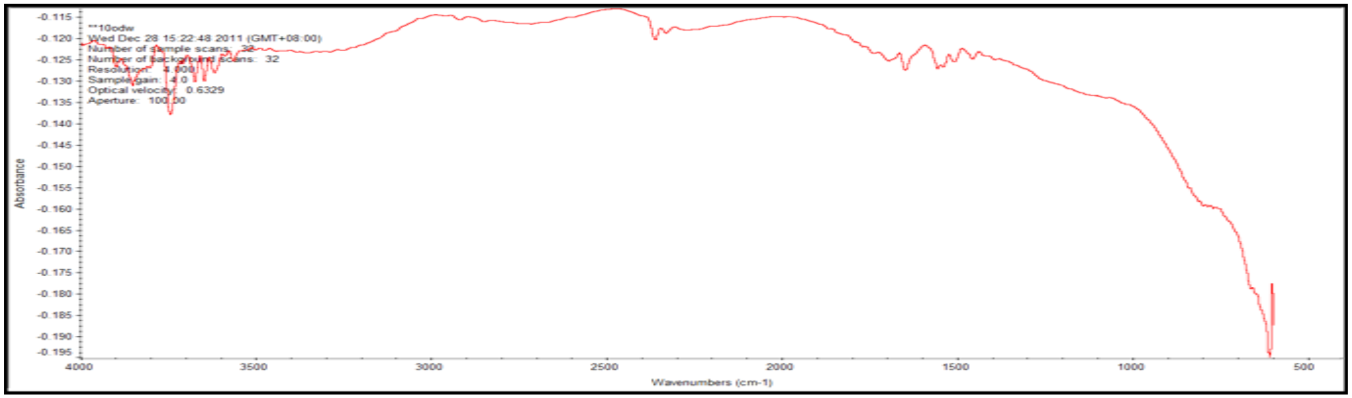


Figure 4.12 Graph of FTIR sample 10

Sample 11;

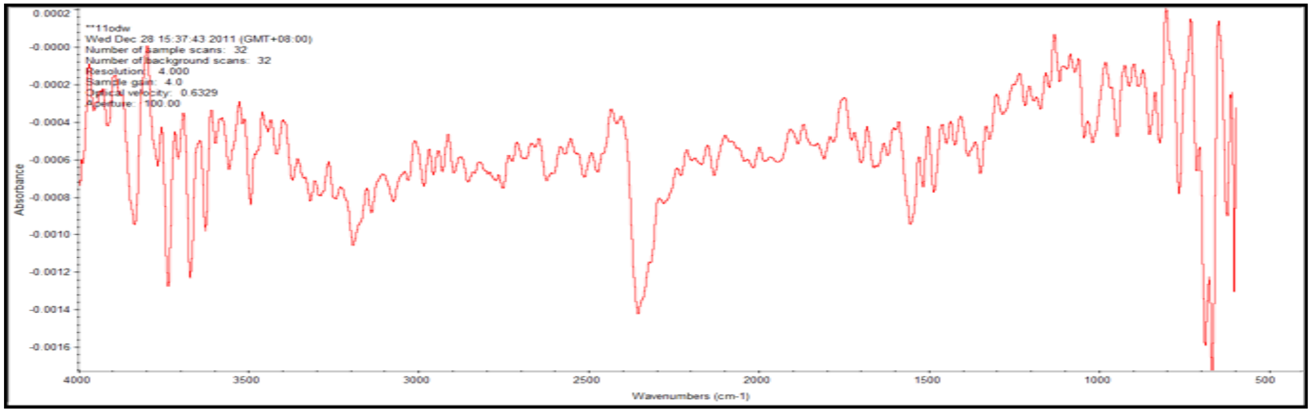


Figure 4.13 Graph of FTIR sample 11

Sample 12;

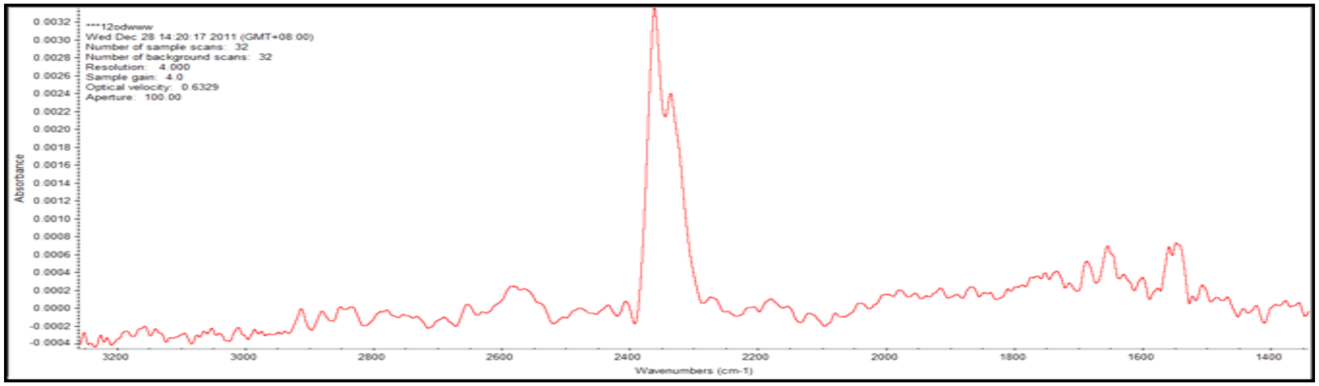


Figure 4.14 Graph of FTIR sample 12

Table 4.6: Functional group found in the mixture

Sample no	Wave no	Functional group	Molecular motion
1	1057.89	sulfoxides	S=O stretch
	1270.15	sulfonates	S=O stretch, S-O stretch
2	2922.01	alkanes	C-H stretch
	2851.22	alkanes	C-H stretch
3	2358.28	Alkynyl	C=C Stretch
	3688.02	Amide	N-H Stretch
	3747.46	Alcohol/Phenol	O-H Stretch
4	662.94	Alkyl halide	C-Cl stretch
	801.65	Amines	N-H bend (oop)
5	2359.54	mercaptans	S-H stretch
	2337.95	mercaptans	S-H stretch
	1062	phosphines	PH bend
	1035.87	sulfones	S=O stretch
6	1652.51	imines	R_2C=N-R stretch
	1698.73	imines	R_2C=N-R stretch
	851.87	aromatics	C-H bend (para)
7	669.75	Alkyl halide	C-Cl stretch
8	2351.06	carboxylic acids	O-H stretch
	2922.01	carboxylic acids	O-H stretch
	3584.68	Alcohol	O-H Stretch
9	2425.04	carboxylic acids	O-H stretch
10	1500.13	aromatics	C=C stretch
	2503.06	mercaptans	S-H stretch
11	2309.17	phosphines	P-H stretch
	1120.58	ketones	C-C stretch
12	2369.53	phosphines	P-H stretch
	2338.12	phosphines	P-H stretch

From the analysis done by FTIR, several compounds have been found such as sulfoxide, alkanes, alkyl halides, alcohol and imines. Although the component of gaharu itself can’t be ascertain, the existence of alcohol in some of the mixture shows that there might be possibilities of the chemical compound of gaharu such as jinkoh-eremol, agarospirol and jinkohol that were OH group miscible in the distilled water analyze.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSION

Conclusion that is made is based on the objectives of this study that was to study the performance of microwave extraction in extracting gaharu essential oil and the effects of microwave power, and days of gaharu soaked in distilled water to the yield extracted. It have been proven that microwave extraction is suitable in extracting gaharu with acceptably low time comparing to other extraction equipment but the yield is too little and as gaharu itself contains low resin in it, the oil extracted were mostly visible at the wall of equipments.

As the power of microwave increases, the yield obtained increases too. This is because; the cell wall of gaharu is loosening leads to increased interaction between extracting agent and source material in the extraction process. As a result, permeation of the extracting agent will be increased thus leads to effective increase in the yield of gaharu essential oil. The days of soaking has also been proven to be directly proportional to the yield of oil extracted, considering the power of microwave set and other condition.

From the analysis done by FTIR, several compounds have been found such as sulfoxide, alkanes, alkyl halides, alcohol and imines. Although the component of gaharu itself can't be ascertain, the existence of alcohol in some of the mixture shows that there might be possibilities of the chemical compound of gaharu such as jinkoh-eremol, agarospirol and jinkohol that were OH group miscible in the distilled water analyze.

5.2 RECOMMENDATIONS

In order to obtain more yield from the extraction, it is advisable to change the parameter to find the optimum condition for the microwave extraction. Several other parameters to be taken into consideration are extraction time, temperature, and the gaharu condition itself that can be varies by sizes, grade. Other than that, it is advisable to use solvent such as hexane and methanol, ethanol or any other relevant to ensure more oil can be extracted from the gaharu wood.

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