

**EXPERIMENTAL WORK ON THE DIFFERENT CONDITIONS OF  
JASMINE FLOWERS ON THE COMPOSITION OF JASMINE  
ABSOLUTE**

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ABSOLUTE**

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

**Faculty of Chemical & Natural Resources Engineering  
UNIVERSITI MALAYSIA PAHANG**

JANUARY 2014

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## **SUPERVISOR'S DECLARATION**

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering (Pure).

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## **STUDENT'S DECLARATION**

I hereby declare that the work in this thesis 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.

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## **ABSTRACT**

Essential oil is the botanical extracts from various plant materials. The essential oils nowadays had been commercialized due to its thousands of benefits. On the other hand, Jasmine flowers had been known for its fragrance value for ages. Jasmine primarily used in perfumery industry because of its therapeutic properties. Basically, there are two types of Jasmine Essential oils, named Jasmine Concrete and Jasmine Absolute. Jasmine Absolute is the pure Jasmine essential oil processed from Jasmine concrete. Same like the other flowers, Jasmine flowers will undergo essential phases of bud, bloom and wilt. In order to get best properties for Jasmine absolute, this thesis will study about the different conditions of Jasmine flowers that will be affected the composition Jasmine absolute. To produce a Jasmine Absolute, method solvent extraction was used as it will give high yield and low cost. In this solvent extraction method, Hexane was used as the solvent. Once the concrete was obtained, it will be mixed with ethanol and then will undergo freezing and distillation process. The properties of each different condition will be then analysed using GC analysis. Based on the analysis, the dry condition give the best qualitative analysis meanwhile the bloom condition give the best quantitative analysis



## **ABSTRAK**

Minyak pati adalah ekstrak botani daripada pelbagai bahan tumbuhan. Minyak pati kini telah dikomersialkan kerana beribu-ribu manfaat. Bunga Jasmin telah dikenali untuk nilai wangian untuk peringkat umur. Jasmin terutamanya digunakan dalam industri minyak wangi kerana sifat terapeutiknya. Pada dasarnya, terdapat dua jenis Jasmine Minyak pati, bernama Jasmine Konkrit dan Jasmine mutlak. Jasmine absolute adalah jasmine minyak tulen penting diproses dari Jasmine konkrit. Sama seperti bunga lain, bunga Jasmine akan menjalani fasa penting dalam putik, mekar dan layu. Dalam usaha untuk mendapatkan sifat-sifat terbaik untuk Jasmine absolute, tesis ini akan mengkaji tentang keadaan yang berbeza bunga Jasmine yang akan menerima kesan komposisi Jasmine absolute. Untuk menghasilkan jasmine absolute, kaedah pengekstrakan pelarut digunakan kerana ia akan memberikan hasil yang tinggi dan kos rendah. Dalam kaedah pengekstrakan pelarut ini, Heksana telah digunakan sebagai pelarut. Apabila konkrit telah diperolehi, ia akan dicampur dengan Etanol dan kemudian akan menjalani proses pembekuan dan penyulingan. Sifat-sifat setiap keadaan berbeza akan dianalisis menggunakan analisis GC. Berdasarkan analisis Keadaan kering memberikan analisis kualitatif yang terbaik Sementara itu keadaan mekar memberikan analisis kuantitatif yang terbaik.

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## **LIST OF ABBREVIATIONS**

HPLC	High Performance Liquid Chromatography
L	liter
mg	milligrams
min	minutes
ml	milliliter
nm	nano metre
ppm	Part per million, mg/L

## **LIST OF APPENDICES**

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## LIST OF SYMBOLS

$^{\circ}\text{C}$	Degree celcius
$C_e$	Equilibrium concentration
$C_o$	Initial concentration
$K_f$	Freundlich constant
$V$	Volume



## **CHAPTER ONE**

### **INTRODUCTION**

#### **1.1 Background of The Proposed Study**

Essential oil is a liquid that generally distilled from leaves, stems, flowers, bark, roots or other elements in plants. It usually highly concentrated, volatile and has high aromatic essences of plants. The essential oils are clear and contain the true essence of the plant where it derived from. Essential oils from flowers are widely used in perfumery industries and have a very high commercial value. A plant produces its essential oil because of the protoplasm in the cells. The oil itself is actually the excretion which not involve in metabolism of the plant. The oils high in energy and chemically active, hence it is surprising for the plant to expel the energy without use it. The oils are stored in the form of micro duplets in the glands of the plants. After diffusing slowly through the wall of the gland, the droplets spread out on the surface of the plants before evaporating and filling the air with the perfume. Among flowers, the most attractive perfume is jasmine flowers. Jasmine essential oil named Jasmine concrete. Jasmine concrete is not a pure

essential oil of jasmine flowers since it contains impurities. Jasmine absolute is a type of pure jasmine essential oil. Jasmine flowers or scientifically known as *Jasminum grandiflorum* is known for century but very recently extraction of essential oil known as 'Jasmine absolute' has been done for large scale (National Institute of Industrial Research, 2007). Same like to other common flowers, jasmine can be exists in form of bud, bloom and dry. This research was conducted in order to figure out the best jasmine flower condition to the highest absolute qualities. In few cases, raw cosmetic materials are treated at early stage to avoid degradation for their quality. When the raw materials undergo solvent vaporization, it gives a product called 'concrete'. Research done by Olesegun, E (2012), 'concrete' contains fragrances compounds such as hydrocarbon terpenes, oxygenated terpenes, sesquiterpenes, oxygenated sesquiterpenes, and other flavoring compounds.

For this thesis paper, the extraction process used to be conducted is solvent extraction. Among most of flowers, jasmine is one of the most attractive flowers. This research was undertaken to identify the best flower's condition which are bud, bloom and dry in order to produce jasmine absolute.

## **1.2 Problem Statement**

For this research, jasmine flower is being used as substrate. *Jasminum officinalis*, or also known as Melur is commonly extracted for its essential oils. The aroma of jasmine is potently sweet and floral, hence it is a favorite with almost everyone. The flowers are delicate and only open during evening. Jasmine petals usually remain open between six and eight during evenings when the temperature comes down.

Generally, there are many factors to be taken into consideration when producing good quality essential oil, which is crucial for its full benefit. According to (Huei, 2002) the factors affecting the composition of essential oils are climate, soil condition, air quality, cultivation, harvesting, extraction method used, storage, transportation conditions and age of the oil. Based on his research, it is showed that harvesting plays an important role. In harvesting, conditions of flowers been harvested also plays an important role. Same to the other typical flowers, jasmine

undergo phase of bud, bloom and dry. In order to produce the best jasmine essential oil, the best condition for the flower's condition was being studied.

In Malaysia, Jasmine flower is commonly used by the Indians during the Diwali celebration. Jasmine is important traditional flower cultivated in India. However, the needed of jasmine flower is decreasing during the non-Diwali time. Hence, the flowers were being unused. It will be a waste for a material with high therapeutic value. As for its own commercial value, this research will be an advantage added to the essential oil's entrepreneur in Malaysia.

Jasmine flower contains very little volatile oil, hence it needed to be solvent extracted since it was too delicate to be distilled. For this research, solvent extraction is the most suitable method as it will produce high yield and low cost. These three conditions will be compared to identify which condition with highest purity using Gas Chromatography (GC) analysis.

### **1.3 Research Objective**

The specific objectives of this study include:

1. To extract the jasmine flowers
2. To analyzed the samples components by using Gas Chromatography (GC).
3. To obtain the optimum condition of highest quality and yield of jasmine absolute.

### **1.4 Scope of The Proposed Study**

In order to achieve the objectives, the scopes of studies were identified as follows:

1. Preparation of Sample
  - Jasmine flowers was collected
  - The flowers was separated according to different conditions which are bud, bloom and wilt

- After that, jasmine flowers were blended.

## 2. Extraction Process

- The jasmine flowers were extracted by using solvent extraction. The solvent extraction was done twice using two different chemicals. The first chemical is Hexane and the second chemical is Ethanol.

## 3. Analysis

The extraction yields were analysed by using Gas Chromatography (GC) to identify the presence of Benzyl Benzoate and Benzaldehyde which are the main constituents of jasmine absolute.

## **CHAPTER TWO**

### **LITERATURE REVIEW**




#### **2.1 Overview**




This chapter will be focused on the review of jasmine flowers which is the substrate of this project, followed by the extraction process of the essential oils that were involved in this study will be reviewed Other than that, the information of jasmine essential oil which is the product of this study. The aim of this chapter was to gather all the information that can contribute towards this research.

Jasmine is a type of flowers that derived from Persia ‘Yasmine’ which means gift from god. Jasmine plants are native to tropical, subtropical and warm areas of Europe, Asia, Africa and Australia. Jasmine flowers are very well known around the world because of its fragrance. The true jasmine grows as climbing vine with oval, shiny leaves and tubular waxy white flowers. There are many types of Jasmine flowers. The most popular jasmines are *Jasminum humile*, *Jasminum nitidum* *Jasminum officinale* *Jasminum sambac*. According to Sheela (2008), Jasmines are the most common flowers in India garden.

According to D.Ravinath (2007) ,There are many uses of Jasmine flowers. They are usually used as pot plants. Other than that, because of their fragrance, they have been used for making garlands. Jasmine flowers also been used widely in perfumery industry. Jasmine essential oil is high market value due to its property.

**Table 2.1:** The types of jasmine flowers

Type of Jasmines	Description
<p data-bbox="298 569 531 600">Jasminum humile</p> 	<p data-bbox="667 569 1289 709">Yellow-flowered plant that has a nice fragrance and can bloom almost any time a year. It can tolerate a cool green-house nicely.</p>
<p data-bbox="298 989 531 1020">Jasminum nitidum</p> 	<p data-bbox="667 989 1289 1241">Good container plant with bushy green leaves and star-shaped flowers. It can bloom in the cooler months as well as in summer and can tolerate a very cool greenhouse as long it doesn't get below freezing.</p>
<p data-bbox="285 1409 544 1440">Jasminum officinale</p> 	<p data-bbox="667 1409 1289 1549">Common jasmine. It is a slow growing vining plant with fragrant white flowers that can appear almost anytime except late winter.</p> <p data-bbox="667 1570 1289 1661">Commonly used in manufacture perfume in France.</p>
<p data-bbox="269 1829 560 1860">Jasminum polyanthum</p>	<p data-bbox="667 1829 1289 1860">Fast growing plant that has finely divided</p>

	<p>leaves that like to vine around other near plants or structures. It is primarily a prolific winter bloomer, described as blizzard f blooms.</p>
<p>Jasminum sambac</p> 	<p>Commercially used in making tea and perfume. Also important in Buddha ceremonies. It required bright sunlight.</p>
<p>Jasminum tortuosum</p> 	<p>Good choice for a year-round bloomer but blooms heaviest from spring through fall. It is relatively grow faster and has a vining habit. It can tolerate with a wide range of temperature.</p>

## 2.2 Types Extraction of Jasmine Flowers

There are many types available for the extraction of Jasmine flowers. However, all the methods had its own pros and cons. Volatile oils can be recovered from plants by different methods either in conventional technique or new technique. (Kar. A.,2003).

## **2.2.1 Conventional Techniques**

### **2.2.1.1 Extraction with volatile solvent.**

The plant material containing the volatile oil is usually extracted with a low boiling volatile solvent, such as hexane, benzene, petroleum ether and etc., either by adopting method of continuous extraction (Soxhlet extraction) by percolation. The resulting volatile oil containing solvent is removed under reduced pressure when volatile will remain in the flask.

There are several advantages of this process:

1. It is possible to maintain uniform temperature (usually 50°C) during most of these extractions which ultimately ensures the retention of more intense and natural fragrance which otherwise cannot be achieved by distillation (perhaps due to chemical degradation of constituents)
2. Floral Concretes: The ultimate concentrated and purified volatile oils are collectively designated as 'floral concretes'. In actual practice, these floral concretes represent an admixture of natural odoriferous components of flowers, plant waxes, colour pigments and certain albuminous material. Hence, most of them are solid in consistency and partly soluble in 95% alcohol.

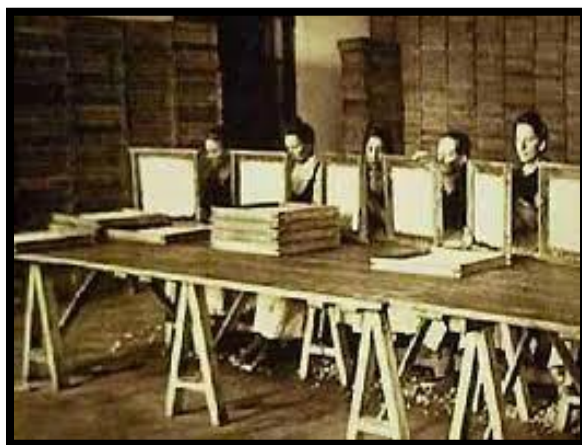
### **2.2.1.2 Extraction with Non-Volatile Solvents.**

The process is usually employed for the preparation of the finest brands of perfume oil, for example the natural flower oils. In this instance, the volatile oil content usually present in fresh plants for example of flower petals. It is so scanty that oil removal is not commercially viable by any other methods.



### **2.2.1.2.1 Enfleurage Method**

A thick layer of molten lard and tallow (beef flat) is applied on either surfaces of pre-cleaned glass plates that are securely placed in a covered wooden fram (or the chasis). Each glass plate is liberally sprinkled with fresh flowers petals to cover its top surface only. These plates are now stacked one over the other enclosed in wooden frame, whereby each layer of flower shall be enclosed between two layers of fat. Such batteries loaded plates are allowed to remain for 24 hours, after which flowers are removed and recharged with fresh lots. This very process is repeated religiously for several weeks till the fatty layers appears to be fully saturated with essential oils of flowers or until certain desired concentration.



**Figure 2.1:** Enfleurage method.

### **2.2.1.2.2 Pneumatic Method**

The basic principle of this method is like enfleurage method. In this particular instance, the current of warm-air is made to pass the flowers, and the subsequent air loaded with suspended volatile oil particles is then routed through a fine spray of molten fat in a closed chamber wherein the volatile oil gets absorbed promptly.

### 2.2.1.2.3 Maceration Method

The fresh flower petals are gently and carefully molten in fat (lard, tallow or fixed oil) stirred frequently until complete exhaustion take place. The flowers are then strained, squeezed and the exuded fat is returned to the main bulk of fat, unless and until a desired concentration is achieved. The volatile oil containing fat is allowed to cool and is recovered by three successive extractions with absolute alcohol.



**Figure 2.2:** Maceration method

## 2.2.2 New Techniques

Microwave-assisted extraction (MAE) together with both Super Critical CO<sub>2</sub> and Continuous Subcritical Water Extraction (CSWE) are considered as the alternatives for highly valuable essential oils.

### 2.2.2.1 Super Critical CO<sub>2</sub>

In this method, carbon dioxide is kept under high pressure at a constant pressure. Plants are placed in a stainless steel tank and, as carbon dioxide is injected into the tank, pressure inside the tank builds up. Under high pressure, the carbon dioxide turns into liquid and act as solvent to

extract essential oils from the plants. When pressure is decreased, the carbon dioxide returns to gaseous state, leaving no residues behind. The equipment for this process is very expensive and so resulting oils. CO<sub>2</sub> extraction process avoid heat degradation to the plant matter, producing an essential oil that is more authentic version of original plant matter (Scalia, S. et al, 1999)

#### **2.2.2.2 Microwave Assisted Extraction**

This is a simple technique that provides a novel way of extracting soluble products into a fluid, from wide range of materials, helped by microwave energy. The first method extraction of essential oil from plant products (Scalia, S. et al, 1999)

### **2.3 Introduction to Jasmine Essential Oils.**

Essential oils are the volatile oils distilled from aromatic plant materials. The odor and flavour of the oils is usually depend on the oxygenated compounds. Essential oils can be divided into two categories which are large volume oils. Large volume oils are usually distilled from leafy materials. The other category is small volume oils which usually distilled from fruits, seeds, bud and flowers. Hence, Jasmine essential oil is a type of small volume oils.

As research done by Abdelouaheb Djilani and Amadou Dicki (2002) shows that essential oils are highly complex mixture of volatile compouns and many contain about 20 to 60 individual compounds, albeit some may contains more than 100 components such as jasmine , lemon and cinnamon essential oils.

Absolute jasmine is a yellowish brown viscous oily substance having delicate odor of fresh Jasmine flowers. The yield of the absolute 40-60% depends on the quality of the concrete.

Properties of absolute jasmine properties is varies depending on several factors. Many factors affected chemical composition hence changing therapeutic value of essential oil. According to study done by (Huei, 2002) , the factors that affecting composition are the chemical

composition depend on climate, soil condition, air quality, cultivation, harvesting, extraction method being used, storage, transportation conditions and age of the oil. As research done by Huei, it is showed that harvesting will affected the properties of absolute Jasmine. So, this research will identified the best flower conditions for the best properties of Jasmine absolute

## **2.4 The Application of Jasmine Essential Oil.**

There are thousands of unexpected benefits of Jasmine essential oil. They are either in odorants, flavors and pharmaceutical. Jasmine may help the body recover from breathing difficulties, bronchial spasms, coughs, depression, dermatitis, eczema, insect bites, labor pains, laryngitis, low self-confident, nervous, pre natal depression, postdated enlargement, skin irritation, stretch mark and sunburn. Based on book written by, when inhaled, jasmine has prodound effect on the wave patterns. Innocence, sweetness and purity are the key inner emotions awakened those who smell jasmine. Jasmine symbolizes hope, happiness and love. Jasmine captivatong scent sedates the nervous system. It reduces anxiety, severe depression and diminishes fear. It soothes the body, mind and spirit for those exposed to emotional abuse and violence. As research done by (Medforth *et al*,2011) Jasmine essential oil can be used as aromatherapy that been used traditionally that aid childbirth. It helps to strengthen the contractions and also act as pain relieving and antispasmodic. It is also emotionally energizing and has antidepressant qualities. Jasmine oil has many uses. It stimulates the immune system, destroying bacteria, and balancing for dry or oily skin. Other than that, Jasmine essential oil can help with laryngitis, immune system and with feminine issues (Jones, 2011).

## **2.5 Types of Extraction**

There are several types for extraction process including:

### **1. Solid – Liquid extraction**

- The principle for the solid-liquid extraction is that the soluble compounds of a solid matter, existing of an inert matrix and the active agent, are extracted by a solvent.

The extract can be included in the extraction matter in solid or liquid form. It can be included in cells like oil in oil seeds or as fine dispersion on the solid matter like caffeine in coffee.

## 2. Liquid – Liquid Extraction

- Liquid - liquid extraction also known as solvent extraction. Theoretically, it is a method to separate compounds based on their relative solubilities in two different immiscible liquid. After extraction the feed and solvent phases are called the raffinate (R) and extract (E) phases respectively.

### **2.6 Rotary Extraction**

Rotary extraction (Figure 2.11) is one of the oldest method and most widely used approaches for conventional extraction of solid samples. It has been used for a long time, is a standard technique and the main reference for evaluating the performance of other liquid–liquid extraction methods. Soxhlet extraction is a general and well-established technique, which surpasses in performance other ordinary extraction techniques (Macfarlane and Cullen, 1998)



**Figure 2.3** Rotary Extractor

### **2.6.1 Advantages of Rotary Extractor**

The advantages of this process are:

1. No filtration process is required after extraction process complete.
2. Simple, cheap and easy to operate. (Machell 1994).
3. The compounds are not decomposed due to moderate extraction conditions.

## **2.7 Parameters Affect Solid-Liquid Extraction**

### **2.7.1 Solvent Selection**

The solvent is the important parameter to achieve successful separation by solid-liquid extraction. A solvent can be describe as liquid or gas which dissolves solid, liquid or gaseous solute to form a solution

There are several criteria that influence the selection of solvent:

- Density

- A big difference in density between extract and raffinate phases lead to high capacities in equipment. This is especially important for extraction devices utilizing gravity for phase separation
- Selectivity
  - If there are more than one solutes (solute A and solute B), then consideration should be given to the selectivity of the solvent for solute A as against B. The selectivity between the 2 solutes A and B is defined as the ratio of the distribution coefficient of A to the distribution coefficient of B (Wallis *et al.*, 1997).
- Chemical reactivity
  - The solvent should be stable chemically and inert toward the other components of the system and toward the common materials of construction.
- Availability and cost
  - The solvent must be cheap and easily available.

Solvent can be classified into three categories which is polar protic, polar aprotic and non-polar. Basically, the dielectric constant of the solvent provides a rough measure of a solvent's polarity.

### 2.7.2. Polar protic solvents

Polar protic solvents tend to have high dielectric constants and high dipole moments. Furthermore, since they possess O-H or N-H bonds, they can also participate in hydrogen bonding. These solvents can also serve as acids which is the sources of protons and weak nucleophiles which forming bonds with strong electrophiles.. They are most commonly used as the solvent for their conjugate bases (Christian and Thomas, 2011). Figure 2.13 shows the examples of polar protic solvents:

Polar Protic Solvents		Dielectric constant	Dipole Moment
$\text{:NH}_3$	<i>Ammonia</i>	~25	1.4 D
$\begin{array}{c} \text{CH}_3 \\   \\ \text{H}-\ddot{\text{O}}-\text{C}-\text{CH}_3 \\   \\ \text{CH}_3 \end{array}$	<i>t-Butanol</i>	12	1.7 D
$\text{H}-\ddot{\text{O}}-\text{CH}_2\text{CH}_2\text{CH}_3$	<i>n-Propanol</i>	20	1.68 D
$\text{H}-\ddot{\text{O}}-\text{CH}_2\text{CH}_3$	<i>Ethanol</i>	25	1.69 D
$\text{H}-\ddot{\text{O}}-\text{CH}_3$	<i>Methanol</i>	33	1.70 D
$\begin{array}{c} \text{O} \\    \\ \text{H}-\ddot{\text{O}}-\text{C}-\text{CH}_3 \end{array}$	<i>Acetic acid</i>	6.2	1.74 D
$\text{H}-\ddot{\text{O}}-\text{H}$	<i>Water</i>	80	1.85 D

**Figure 2.4** Polar protic solvents

Ethanol known as ethyl alcohol can be describe as volatile, flammable, colorless liquid. It is a solvent that miscible with water including with many organic solvents. It is also miscible with light aliphatic hydrocarbons such as pentane and hexane, with aliphatic chlorides such as trichloroethane and tetrachloroethylene (Eric, 2006). It is found used in alcoholic beverages and fuel.



**Table 2.2** Physical and chemical properties of ethanol

<b>Properties of Ethanol</b>	
Molecular formula	C <sub>2</sub> H <sub>6</sub> O
Molar mass	46.07g/mol
Density	0.789g/cm <sup>3</sup> (at 20 °C)
Boiling point	78.4°C
Melting point	-114°C
Specific gravity	0.79
Vapor density	1.49
Vapor pressure	5.95kPa (at 20 °C)

N-hexane is a very volatile aliphatic hydrocarbon. It is a constituent in the paraffin fraction of crude oil and natural gas and is also used as an industrial chemical and laboratory reagent. Laboratory grade *n*-hexane contains approximately 99% *n*-hexane. “Hexane” or “hexanes” is a commercial and industrial product consisting of a mixture of hydrocarbons with six carbon atoms and includes *n*-hexane and its isomers 2-methylpentane and 3-methylpentane as well as small amounts of other hydrocarbons (Brugnone et al.1991).

**Table 2.3** Physical and chemical properties of n-Hexane

<b>Properties of n-Hexane</b>	
Molecular formula	C <sub>6</sub> H <sub>14</sub>
Molar mass	86.18g/mol
Density	0.692g/cm <sup>3</sup> (at 20 °C)
Boiling point	98°C
Melting point	-114°C
Specific gravity	0.79
Vapor density	3.5
Vapor pressure	40mm Hg (at 20 °C)

### **2.7.2 Mass of material**

The other factor that affects the rate of extraction process is the mass of raw material (gram). Basically, by increasing the mass of material, the surface contact between solid phase and liquid phase will be increase. Thus, more solute can be obtained and higher yield will be produce.

### **2.8 Summary**

For this study, there will be solvent extraction using Ethanol and n-hexane. The mass of the raw material will be used is 1000g.

## **CHAPTER 3**

### **MATERIALS AND METHODS**

#### **3.1 Overview**

This chapter will be discuss about the raw materials being used in this project including the preparation, methods to prepare and the steps of how this project was being conducted. The project was divided into two parts, the extraction of Jasmine flowers by using solvent extraction and analysis by using Gas Chromotography (GC).

## **3.2 Apparatus and Equipment**

### **3.2.1 Apparatus**

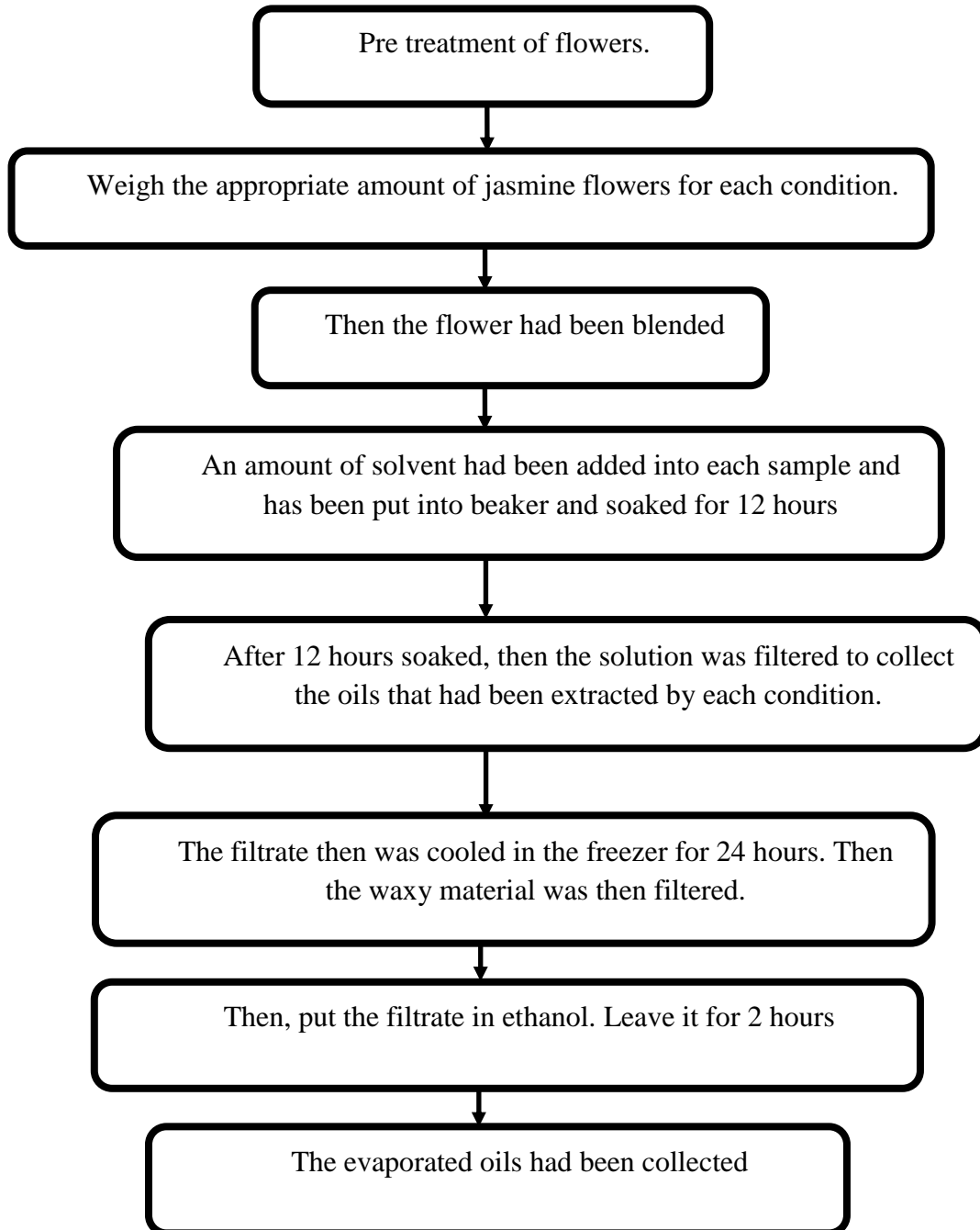
- a) 10 ml, 100 ml and 250 ml beaker
- b) Conical flask 250 ml
- c) Volumetric flask 100 ml
- d) Parafilm
- e) Labeling sticker
- f) Spatula
- g) Tissue paper
- h) Aluminum foil
- i) Funnel
- j) Analytical balance (B204-S Mettler Toledo, USA)

### **3.2.2 Equipment**

- a) Gas Chromatography (GC)
- b) Rotary Evaporator
- c) Drying oven

### 3.3 Methods

#### 3.3.1 General Methodology



**Figure 3.1** Process flow diagrams of procedures

### 3.3.2 Preparation of raw material

The raw material for this project is jasmine flowers. The materials were purchased at the local market which is located in Kuantan. The jasmine flowers were divided into 3 parts; bud, bloom and dry. As for bud conditions, the flowers were stored into the freezer, bloom was left on the table to let it bloom, meanwhile for dry, the flowers was left in the drying oven for 2 days. This to ensure all the moisture content is completely evaporated. The bud jasmine flowers (Figure 3.2) were grounded in domestic blender to increase the surface area and thus, speed up during the extraction process. The same steps were repeated to bloom and dry flowers.



**Figure 3.2:** Jasmine flowers were blended into small pieces

### 3.3.3 Preparation of solvents

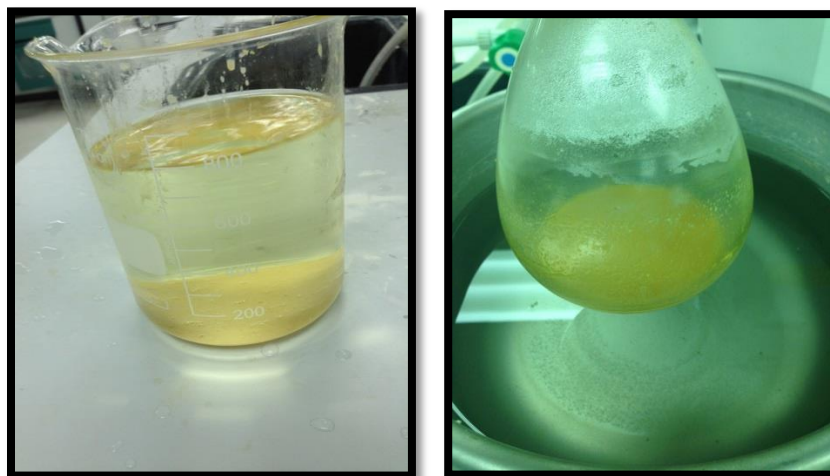
Solvent that will be used for extraction process n-hexane and ethanol (Figure 3.3).



**Figure 3.3** n-Hexane (95%) and Ethanol.

### 3.3.4 Solvent Extraction

Jasmine flowers with amount of 1 kg were placed in a beaker. Then, 2L amount of 95% n-hexane was put into the beaker. The beaker was covered using paraffin film. This is to prevent the hexane exposed to the air as it will cause slightly irritating to the respiratory system (MSDS Hexane). The sample was then left for 24 hours. This to ensure that solvent extraction process had maximized. After that, the sample was then filtered using conventional filter. The filtrate is the mixture of n-hexane and jasmine essential oil.(Figure 3.4) In order to obtain the oil, rotary evaporator was used.

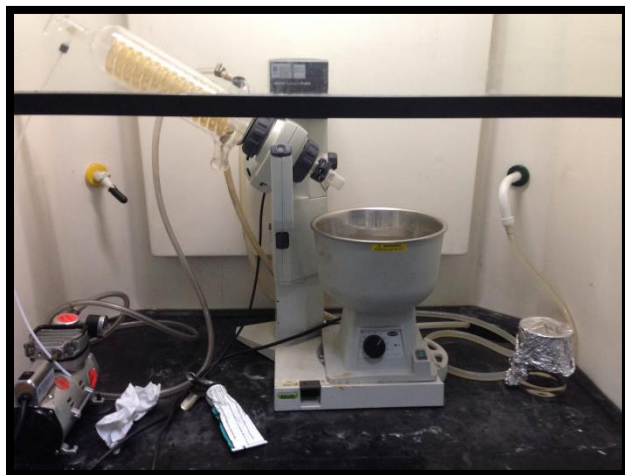


**Figure 3.4** Filtrate as to become Concrete.

### **3.3.5 Evaporation of solvent**

After the extraction process completed, the rotary evaporator was used to remove the solvent based on their boiling point. (Figure 3.6) For this process, in order to remove Hexane, temperature was set at 68 °C. Typical rotary evaporator has a water bath that can be heated in either a metal container or crystallization dish. This keeps the solvent from freezing during the evaporation process. The solvent is removed under vacuum, is trapped by a condenser and is collected for easy reuse or disposal. After the solvent was completely removed from the sample, a small quantity of liquid residue remained inside the bottom flask. This is called Jasmine Concrete. Jasmine concrete was measured using measuring cylinder and the recovered hexane was stored for the next conditions. Jasmine concrete was stored under refrigeration until used for next step.





**Figure 3.5** Rotary Evaporator

### **3.3.6 Solvent Extraction (Ethanol)**

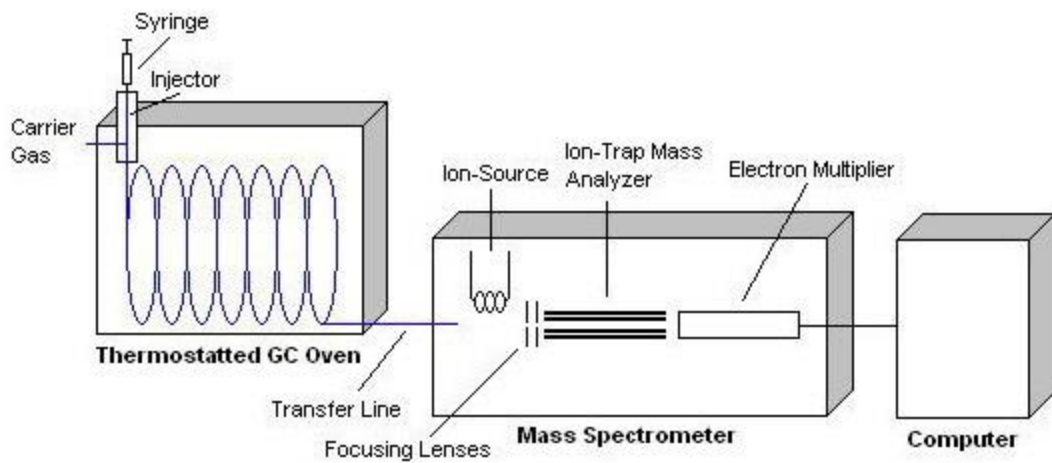
The concrete then was mixed with Ethanol to ratio 1:3. The mixed solution then was left in the freezer at temperature 0-4 degree. This is to remove the waxy materials. The solution was left in freezer for 12 hours. After that, filter the solution and the mixed of Ethanol and Jasmine Absolute was obtained. The solution then was put again in rotary evaporator (Figure 3.4) in order to get Jasmine Absolute.



**Figure 3.6** The filtration of waxy materials

### **3.3.6 Analysis**

In order to know the composition of the components of Jasmine Absolute, Gas Mass Spectrometry was used. The composition analysis of the essential oils were carried out using gas chromatography (Agilent Technologies 6890 USA) which was equipped with Mass Spectrometry (GC-MS) , and capillary column. Helium gas was used as carrier gas at 30cm/sec flow rate ad 2.92 psi inlet pressure. Temperature was set up from 40 °C to 260 °C with a final hold time 3 min. Injector and detector was mantained at 250 °C and 300 °C. 1  $\mu$ L of oil samples were injected using a syringe.



**Figure 3.7** Gas Chromatography Mass Spectrometry (GC-MS) for analysis

## **CHAPTER 4**

### **EXTRACTION OF JASMINE ABSOLUTE**

#### **4.1 Overview**

The results obtained in these studies will be discussed details in this chapter. Firstly, it will be discussed about physical characteristic of the Jasmine Absolute. Then, it will follow by the identification of the main components by using Gas Chromotography Mass Spectrometry (GC-MS).

## 4.2 Physical Properties

As for the physical properties, the colour of the jasmine absolute was found to be reddish brown colour (Figure 4). This is similar to the finding found by Gilbert *et al.*, (1999) which found that the colour of jasmine oil is brown reddish in colour. The colour for three conditions was found to be same which are brown reddish. The phenolic extractives contribute the colour of the jasmine absolute. The phenolic extractive such as phenol is in dark brown colours (Dai & Mumper, 2010).



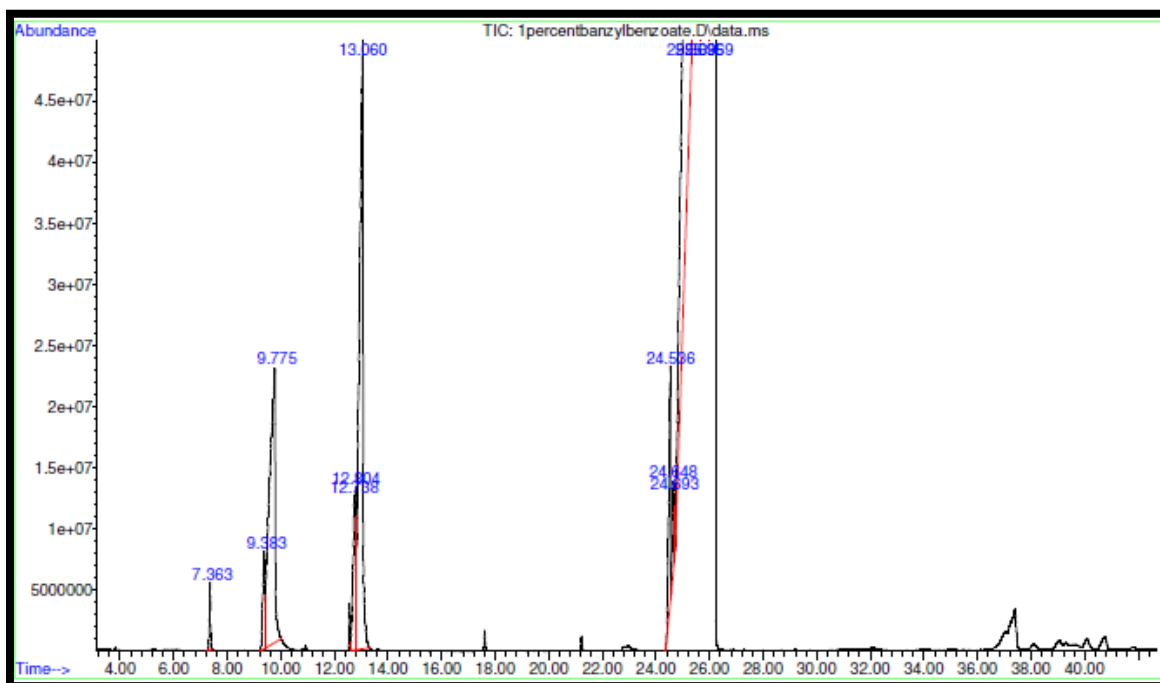
**Figure 4.1:** Jasmine Absolute

**Table 4.1** The physical of Jasmine Absolute

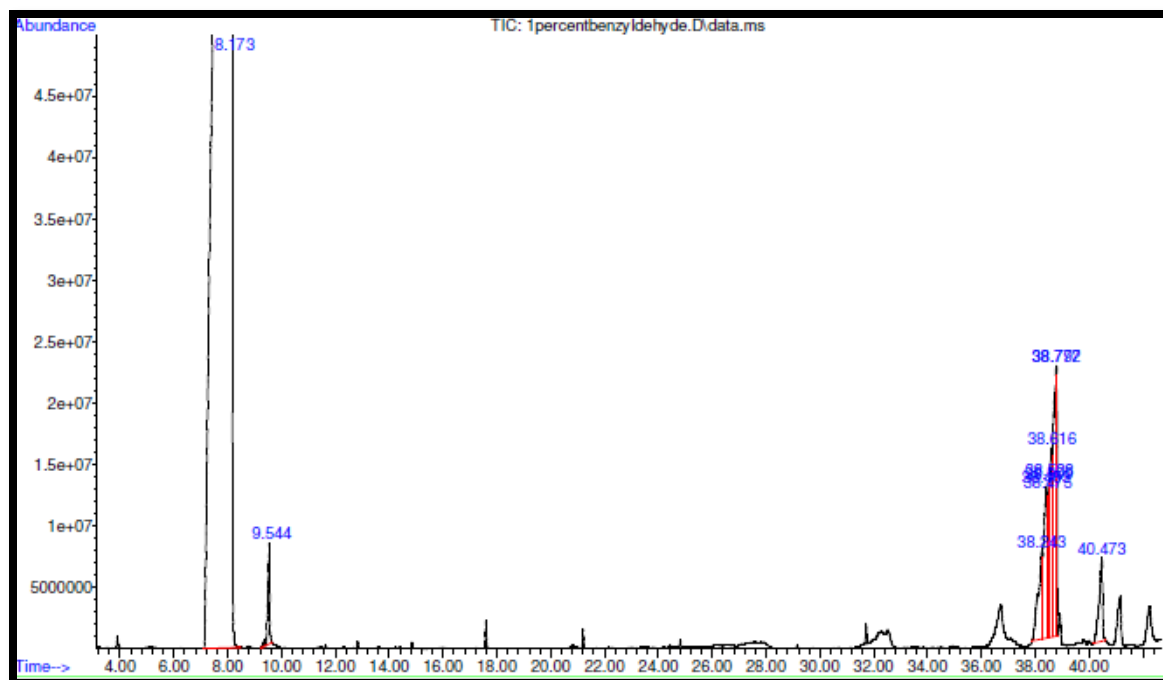
<b>Appearance</b>	Viscous Liquid
Color	Reddish Brown
Solubility	
a) Solvent	Soluble
b) Alcohol	Soluble
c) Water	Insoluble

### 4.3 GC Analysis of Benzaldehyde and Benzyl Benzoate

The jasmine essential oil contains more than 100 constituents, but the components that responsible for the presence of the aroma are benzyl acetate, benzyl alcohol, benzaldehyde, cis-jasmone, geraniol, farnesol and etc ( Lawrence, 1977). Based on chromatogram shown in Figure 4.2, the retention time of Benzyl Benzoate is at retention time 24.543 min. In this analysis of Benzyl Benzoate, there are some other components also exist in Benzyl Benzoate such as Benzyl Alcohol, Benzoic Acid and Benzenecarboxylic acid. In Figure 4.3, component Benzaldehyde was traced at retention time of 8.175 mins, followed by Benzyl Alcohol at retention time 9.543 mins, and other components.



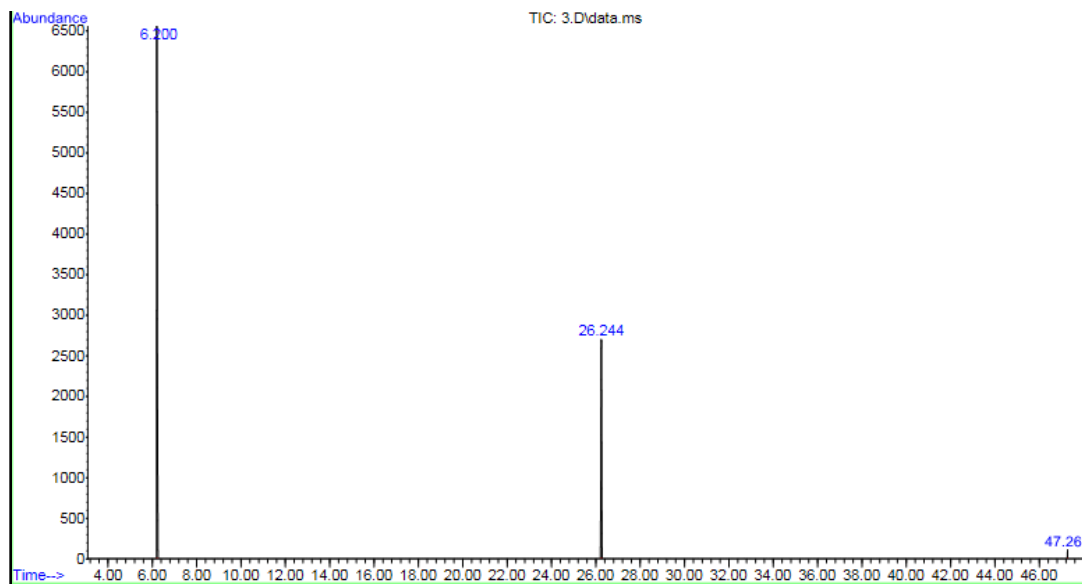
**Figure 4.2** GC Analysis of Benzylbenzoate



**Figure 4.3:** GC Analysis of Benzaldehyde.

### 4.3.1 GC Analysis of Bud Absolute

Based on the chromatograph, it was found that the retention time 6.2 mins, the first peak which is Benzaldehyde was found. The peak height is 6559. At retention time 26.2438 mins, third peak which is the Benzyl Benzoate was found with height 111.

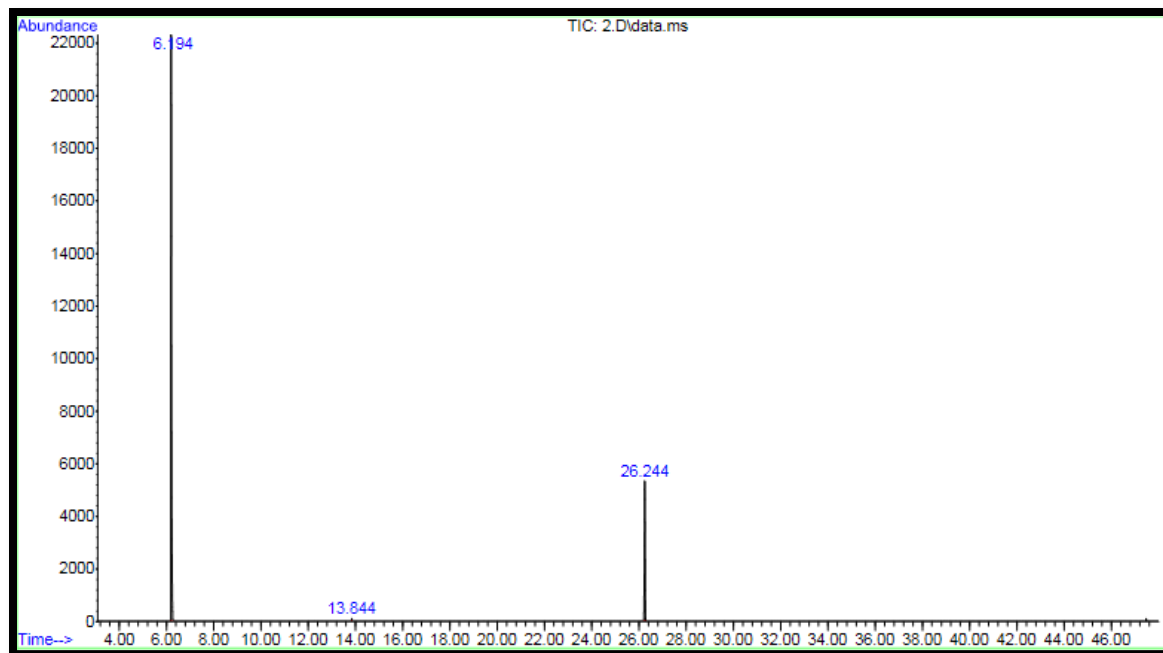


**Figure 4.4:** GC Analysis of Bud Absolute.

### 4.3.2 GC Analysis of Bloom Absolute

Based on the chromatograph, it was found that the retention time 6.194 mins, the first peak which is Benzaldehyde was found. The height area is 22321. At retention time 26.244mins, third peak which is the Benzyl Benzoate was found with height area of 5346.

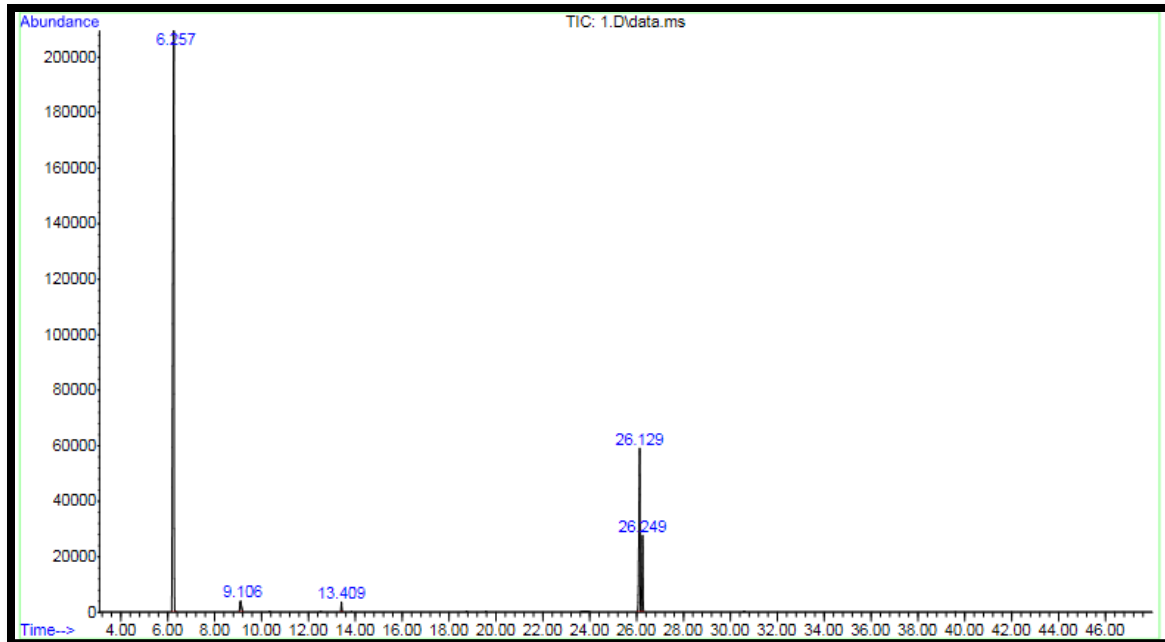




**Figure 4.5:** GC Analysis of Bloom Absolute.

### 4.3.3 GC Analysis of Dry Absolute

Based on the chromatograph, it was found that the retention time 6.257 mins, the first peak which is Benzaldehyde was found. The height of this peak is 209551. At retention time 26.129mins, third peak which is the Benzyl Benzoate was found. The peak height is 59133.



**Figure 4.6:** GC Analysis of Dry Absolute

#### 4.3.2 Comparison of Peak Height between Flower Conditions

According to (Lawrence, 1977) the main chemical components responsible for aroma is Benzyl Acetate, Linalool, Benzyl Alcohol, Benzaldehyde and etc. Hence, the comparison peak height for the Benzaldehyde was figured out. Based on the comparison, it is clearly shown that the dry absolute has the highest peak. Hence, qualitatively, bud absolute has the best condition. In this chromatograph, other components cannot be traced. This is maybe due to the denature during heating. Hence, dry flower condition is the optimum flower condition in order to produce jasmine essential oil. According to Wallis *et al.*, (1997), desired moisture content must be at below of 10 % prior to the process in order to get the optimum result.

#### 4.4 Comparison of Jasmine Absolute's Yield

**Table 4.2** Effect of various parameters on yield ( Concrete)

<b>Condition</b>	<b>Weight Before Extracted (ml)</b>	<b>Weight After Extracted (ml)</b>	<b>Percentage Yield (%)</b>
Bud	1254	56	4.46
Bloom	1246	68	5.45
Dry	1243	40	3.28

**Table 4.3** Effect of various parameters on yield (Absolute)

<b>Condition</b>	<b>Weight Before Extracted (ml)</b>	<b>Weight After Extracted (ml)</b>	<b>Percentage Yield (%)</b>
Bud	1254	8	0.635
Bloom	1246	15	1.203
Dry	1243	5	0.402

Value of Table 4.2 and 4.3 shows the difference yield of concrete and absolute. Based on the result, it can see that the optimum condition of yield for this research is bloom flower condition. With the optimum condition, both jasmine concrete and jasmine absolute produce highest percentage yield. It is generally assumed that the material is best collected when flower have reached their optimal state of development. ( Ghalamreza, et al.,2003). This result similar finding to the Holm et al. which found that the highest percentage of oil is the best during flowering stage.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

The objectives of this study are to extract the different condition of jasmine conditions, to analysis the samples component and to determine the optimum condition of the production of jasmine absolute. There are various methods in production of jasmine absolute. The most common method is by solvent extraction process. Extraction process is the withdrawing of an active agent or a waste substance from a solid or liquid mixture with a liquid solvent. The solvent is not or only partial miscible with the solid or the liquid. By vigorous contact the active agent transfers from the solid or liquid mixture (raffinate) into the solvent (extract). After mixing the two phases are separated which happens either by gravity or centrifugal forces. In this process, jasmine absolute is being extracted from different jasmine flower conditions by using rotary extractor.

During the process, jasmine absolute was extracted by using three different conditions of jasmine flowers which are bud, bloom and dry. Each of the conditions had been undergo different pre-treatments process. During this study, different mass of jasmine flower used for each conditions are 1000g. The bleached solvents from the solvent extraction process were extracted for two times in order to get jasmine absolute. The results show the highest yield of jasmine concrete and jasmine absolute of bloom flower conditions recorded was 5.45% and 1.203%. The yield of concrete is higher compare to absolute because the concrete contains all the waxes particles. The lowest yield of jasmine absolute recorded was 3.238% and 0.402% of jasmine absolute. The best qualitative condition for the jasmine absolute is dry flower condition.

## **5.2 Recommendation**

Result obtained from this research found that dry condition is the optimum for the production of jasmine essential oil. Method used for this extraction is solvent extraction. The solvent used for this extraction process is Ethanol and n-hexane. Since n-hexane is hazardous to the lung system, other solvent such as Methanol or Acetone can be use for solvent extraction. In order to increase the production yield of jasmine absolute, increasing the volume of solvent, the extraction process can produce higher yield of the jasmine absolute until it reached equilibrium point and no jasmine flowers can be extracted anymore.

Apart from that, there are other methods to extract jasmine absolute. One of other methods is supercritical fluid extraction. It is readily available at low cost, it has a low toxicity and reactivity, and it provides a clean alternative to conventional liquid or solid extraction techniques. (Velde, 1994).

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APPENDICES

INSTRUMENT CONTROL PARAMETERS: GCMS

-----

C:\MSDCHEM\1\METHODS\CHECKOUT\farhana.m

Tue Jan 07 12:59:43 2014

Control Information

-----

Sample Inlet : GC

Injection Source : GC ALS

Mass Spectrometer : Enabled

No Sample Prep method has been assigned to this method.

=====

6890 GC METHOD

=====

OVEN

Initial temp: 40 'C (On)

Maximum temp: 325 'C

Initial time: 1.00 min

Equilibration time: 1.00 min



Ramps:

#	Rate	Final temp	Final time
---	------	------------	------------

1	5.00	260	3.00
---	------	-----	------

2	0.0(Off)		
---	----------	--	--

Post temp: 70 'C

Post time: 0.00 min

Run time: 48.00 min

FRONT INLET (UNKNOWN)

BACK INLET (SPLIT/SPLITLESS)

Mode: Split

Initial temp: 250 'C (On)

Pressure: 14.29 psi (On)

Split ratio: 50:1

Split flow: 65.9 mL/min

Total flow: 69.8 mL/min

Gas saver: On

Saver flow: 20.0 mL/min

Saver time: 3.00 min

Gas type: Helium

COLUMN 1

COLUMN 2

(not installed)

Capillary Column

Model Number: Agilent 19091S-433

HP-5MS 5% Phenyl Methyl Siloxane

Max temperature: 325 'C

Nominal length: 30.0 m

Nominal diameter: 250.00 um

Nominal film thickness: 0.25 um

Mode: constant flow

Initial flow: 1.3 mL/min

Nominal init pressure: 14.29 psi

Average velocity: 31 cm/sec

Inlet: Back Inlet

Outlet: MSD

Outlet pressure: ambient

FRONT DETECTOR (TCD)

BACK DETECTOR (NO DET)

Temperature: 280 'C (On)

Reference flow: 20.0 mL/min (Off)

Mode: Constant makeup flow

Makeup flow: 7.0 mL/min (Off)

Makeup Gas Type: Helium

Filament: Off

Negative polarity: Off

SIGNAL 1

SIGNAL 2

Data rate: 20 Hz

Data rate: 20 Hz

Type: test plot

Type: test plot

Save Data: Off

Save Data: Off

Zero: 0.0 (Off)

Zero: 0.0 (Off)

Range: 0

Range: 0

Fast Peaks: Off

Fast Peaks: Off

Attenuation: 0

Attenuation: 0

COLUMN COMP 1

COLUMN COMP 2

(No Detectors Installed)

(No Detectors Installed)

THERMAL AUX 2

Use: MSD Transfer Line Heater

Description:

Initial temp: 280 'C (On)

Initial time: 0.00 min

# Rate Final temp Final time

1 0.0(Off)

POST RUN

Post Time: 0.00 min

## TIME TABLE

Time	Specifier	Parameter & Setpoint
------	-----------	----------------------

### GC Injector

Front Injector:

No parameters specified

Back Injector:

Sample Washes	2
Sample Pumps	6
Injection Volume	1.00 microliters
Syringe Size	10.0 microliters
PreInj Solvent A Washes	2
PreInj Solvent B Washes	2
PostInj Solvent A Washes	2
PostInj Solvent B Washes	2
Viscosity Delay	0 seconds
Plunger Speed	Fast
PreInjection Dwell	0.00 minutes

PostInjection Dwell 0.00 minutes

Column 1 Inventory Number : AB001

Column 2 Inventory Number : AB001

#### MS ACQUISITION PARAMETERS

##### General Information

-----

Tune File : atune.u

Acquisition Mode : Scan

##### MS Information

-----

Solvent Delay : 3.00 min

EMV Mode : Absolute

Resulting EM Voltage : 1812

[Scan Parameters]

Low Mass : 50.0  
High Mass : 550.0  
Threshold : 100  
Sample # : 2 A/D Samples 4  
Plot 2 low mass : 10.0  
Plot 2 high mass : 500.0

[MSZones]

MS Source : 230 C maximum 250 C

MS Quad : 150 C maximum 200 C

END OF MS ACQUISITION PARAMETERS

TUNE PARAMETERS for SN: US02050106

-----

Trace Ion Detection is OFF.

EMISSION : 34.610

ENERGY : 69.922

REPELLER : 26.102

IONFOCUS : 90.157

ENTRANCE\_LE : 19.000

EMVOLTS : 3000.000

Actual EMV : 1811.77

GAIN FACTOR : <Unable to calculate gain factor.>

AMUGAIN : 1969.000

AMUOFFSET : 132.000

FILAMENT : 2.000

DCPOLARITY : 1.000

ENTLENSOFFS : 17.820

MASSGAIN : 69.000

MASSOFFSET : -10.000

END OF TUNE PARAMETERS

-----

END OF INSTRUMENT CONTROL PARAMETERS

-----

## Library Search Report

Data Path : D:\Data\farhana\301220132\  
 Data File : 1percentbenzyldehyde.D  
 Acq On : 30 Dec 2013 14:51  
 Operator :  
 Sample : 1percentbenzyldehyde  
 Misc :  
 ALS Vial : 1 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex  
 Integration Events: ChemStation Integrator - autoint1.e

#	RT	Area%	Library/ID	Ref#	CAS#	Qual
1	8.175	85.24	C:\Database\NIST05a.L Benzaldehyde Benzaldehyde Benzaldehyde	4936 4937 4935	000100-52-7 000100-52-7 000100-52-7	90 90 90
2	9.543	0.89	C:\Database\NIST05a.L Benzyl Alcohol Benzyl Alcohol Benzyl Alcohol	5223 5224 5222	000100-51-6 000100-51-6 000100-51-6	98 96 94
3	38.244	1.65	C:\Database\NIST05a.L Octadecane Heneicosane Tetratriacontane	94930 122436 182859	000593-45-3 000629-94-7 014167-59-0	95 91 91
4	38.403	2.43	C:\Database\NIST05a.L Heneicosane Octadecane Hexadecane, 2,6,10,14-tetramethyl-	122436 94930 113507	000629-94-7 000593-45-3 000638-36-8	97 96 93
5	38.473	0.76	C:\Database\NIST05a.L Octadecane Hexadecane, 2,6,10,14-tetramethyl- Heneicosane	94930 113507 122436	000593-45-3 000638-36-8 000629-94-7	96 95 94
6	38.499	0.54	C:\Database\NIST05a.L Octadecane Heneicosane Hexadecane, 2,6,10,14-tetramethyl-	94930 122436 113507	000593-45-3 000629-94-7 000638-36-8	96 95 93
7	38.528	0.38	C:\Database\NIST05a.L Octadecane Hexadecane, 2,6,10,14-tetramethyl- Tetratriacontane	94930 113507 182859	000593-45-3 000638-36-8 014167-59-0	96 93 91
8	38.617	2.09	C:\Database\NIST05a.L Hexadecane, 2,6,10,14-tetramethyl- Tetracosane Heptadecane	113507 146922 85524	000638-36-8 000646-31-1 000629-78-7	93 93 93
9	38.772	3.73	C:\Database\NIST05a.L Heptadecane Pentacosane Eicosane	85524 153747 113492	000629-78-7 000629-99-2 000112-95-8	96 91 91
10	38.798	0.87	C:\Database\NIST05a.L Tetracosane Heptadecane Hexadecane, 2,6,10,14-tetramethyl-	146923 85524 113507	000646-31-1 000629-78-7 000638-36-8	98 96 93
11	40.472	1.42	C:\Database\NIST05a.L Heptadecane, 3-methyl- Tetratetracontane Tetracosane, 11-decyl-	94943 188836 182862	006418-44-6 007098-22-8 055429-84-0	91 90 87



## Library Search Report

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 Data File : 1percentbenzylbenzoate.D  
 Acq On : 30 Dec 2013 15:42  
 Operator :  
 Sample : 1percentbenzylbenzoate  
 Misc :  
 ALS Vial : 2 Sample Multiplier: 1

Search Libraries: C:\Database\NIST05a.L Minimum Quality: 0

Unknown Spectrum: Apex  
 Integration Events: ChemStation Integrator - autoint1.e

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
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2	9.384	1.61	C:\Database\NIST05a.L Benzyl Alcohol Benzyl Alcohol Benzyl Alcohol	5223 5224 5222	000100-51-6 000100-51-6 000100-51-6	98 96 94
3	9.775	12.97	C:\Database\NIST05a.L Benzyl Alcohol Benzyl Alcohol Benzyl Alcohol	5224 5223 5222	000100-51-6 000100-51-6 000100-51-6	96 96 96
4	12.739	2.36	C:\Database\NIST05a.L Benzoic acid, ethyl ester Benzoic acid, ethyl ester Benzoic acid, ethyl ester	23436 23441 23442	000093-89-0 000093-89-0 000093-89-0	95 95 91
5	12.805	1.81	C:\Database\NIST05a.L Benzoic acid, ethyl ester Benzoic acid, ethyl ester Benzoic acid, ethyl ester	23436 23441 23442	000093-89-0 000093-89-0 000093-89-0	95 95 91
6	13.060	18.48	C:\Database\NIST05a.L Benzoic acid, ethyl ester Benzoic acid, ethyl ester Benzenecarboxylic acid	23436 23441 9580	000093-89-0 000093-89-0 000065-85-0	90 83 64
7	24.534	3.30	C:\Database\NIST05a.L Benzyl Benzoate Benzyl Benzoate Benzyl Benzoate	65860 65862 65863	000120-51-4 000120-51-4 000120-51-4	97 97 93
8	24.649	1.16	C:\Database\NIST05a.L Benzyl Benzoate Benzyl Benzoate Benzyl Benzoate	65862 65860 65863	000120-51-4 000120-51-4 000120-51-4	97 97 93
9	24.693	0.34	C:\Database\NIST05a.L Benzyl Benzoate Benzyl Benzoate Benzyl Benzoate	65862 65860 65863	000120-51-4 000120-51-4 000120-51-4	97 97 93
10	25.310	30.05	C:\Database\NIST05a.L Benzyl Benzoate Benzyl Benzoate Benzyl Benzoate	65861 65862 65864	000120-51-4 000120-51-4 000120-51-4	81 76 64
11	25.635	20.12	C:\Database\NIST05a.L Benzyl Benzoate Benzyl Benzoate (4-Phenylazophenyl)methanol	65862 65861 65711	000120-51-4 000120-51-4 065926-74-1	87 74 62
12	25.968	7.25	C:\Database\NIST05a.L Benzyl Benzoate Benzyl Benzoate	65862 65860	000120-51-4 000120-51-4	90 64