HEAT LOSS ANALYSIS OF HYDRODISTILLATION OF MANGOSTEEN PERICARP

TAN EVON

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Faculty of Chemical & Natural Resources Engineering UNIVERSITI MALAYSIA PAHANG

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ABSTRACT

Mangosteen pericarp essential oil is primarily used in traditional Thai medicine and has a high commercial value due to its therapeutic properties. In this experimental work, hydrodistillation was employed as a common method of extracting essential oil. Furthermore it is economically viable and safe. Gas chromatography- flame ionization detector analysis (GC-FID) was performed for Volatile oils of mangosteen pericarp. The main constituent of the essential oil is ferulic acid. In this research, the heat loss of hydrodistillation process of mangosteen pericarp is discussed. The comparison for heat loss between different parts of Clevenger-type apparatus, such as round bottom flask (RBF) and biomass flask(BF). The heat removed by chamber (C) is 5.81 x 10⁶ J. The heat loss in (BF) and (RBF) are 2.22x 10⁶ J and 2.05 x 10⁵ J respectively. The prevention of heat loss of Clevenger-type apparatus can be improved in future study by carrying out the research in large scale.

ABSTRAK

Pengekstrakan minyak kulit manggis terutamanya digunakan dalam perubatan tradisional Thai dan mempunyai nilai komersil yang tinggi disebabkan oleh ciri-ciri terapinya. Di dalam kajian ini, pengekstrakan minyak kulit manggis dilakukan menggunakan kaedah penyulingan hidro kerana ia adalah kaedah yang paling biasa digunakan untuk mengekstrakan minyak. Selain itu, kaedah ini adalah dari segi ekonomi yang berdaya maju dan selamat. Sample minyak yang didapati daripada kajian ini akan mengunakan analisis gas kromatogrfi-pengesan pengionan api. Komponen utama di dalam minyak kulit manggis yang telah dikenalpasti ialah asid ferulic. Dalam kajian ini, kehilangan haba dalam proses pengekstrakan minyak kulit manggis menggunakan kaedah penyulingan hidro telah dibincangkan. Kehilangan haba antara bahagian yang berlainan di radas clevenger-jenis akan dibandingkan seperti bawah pusingan kelalang dan kelalang biomass. Haba yang dikeluarkan dari chamber ialah 5.81 x 10⁶ J. Kehilangan haba bawah pusingan kelalang dan kelalang biomass adalah 2.22x 10⁶ J dan 2.05 x 10⁵ J masing-masing. Cara mengelakkan kehilangan haba boleh bertambah baik pada masa akan datang dengan membuat kajian yang berskala besar.

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LIST OF ABBREVIATIONS

A area implied in the heat transfer process (m) specific heat of the material (J/g/ $^{\circ}$ C) c efficiency of heater 3 convective heat transfer coefficient(W/m/C) h gram g L Litre power of heater (W) P Q The amount of thermal energy (Joules) time required to heat the water until boiled (s) t T_{in} inlet temperature of condenser ($^{\circ}$ C) outlet temperature of condenser ($^{\circ}$ C) T_{out} ΔT temperature difference is absorbed or liberated in a sensible heating process.

CHAPTER 1

INTRODUCTION

1.1 Background of research

Mangosteen (*Garcinia mangostana* L.) is a tropical fruit available in Southeast Asia. It bears dark-purple to red-purple rounded fruits of 5–7 cm in diameter. The edible portion of fruit (aril) is white, soft with a slightly sour taste (Martin, 1980). The mangosteen pericarp is used as traditional medicine for the relief of diarrhea as well as for treatment of skin wounds and disorders (Mahabusarakam, *et al.*, 1987). In Ayurvedic medicine the pericarp of mangosteen-fruit has wide use against inflammation and diarrhea (Balasubramanian, *et al.*, 1988), and cholera and dysentery (Sen *et al.*, 1980b). Mangosteen has become recently popular as an alternative medicinal product.

Mangosteen pericarp has been shown to contain variety phenolic compounds such as xanthones,tannins and anthocyanins. Xanthones and tannins assure astringency to discourage infestation by insects, fungi, plant viruses,bacteria and animal predation .Anthocyanin also is a food additive E163. The natural pigment of anthocyanins.used as a food additive, it is quite safely in food; create more attractive colours for food products.

This research was conducted to analyze the chemical composition of mangosteen pericarp by using Gas chromatography- flame ionization detector analysis (GC-FID). Currently, there are a few conventional and modern methods of extracting essential oils such as by hydro-distillation (Purseglove *et al.*, 1981), solvent extraction (Chrissie, 1996), supercritical fluid extraction (Kelly *et al.*, 2002) and microwave extraction (Soud *et al.*, 2002). The extraction of essential oil of mangosteen pericarp is using hydrodistillation method as it is the oldest and most common method of extracting essential oil. Furthermore it is economically viable and safe.

Hydrodistillation is made with the Clevenger type apparatus. The mangosteen pericarp is immersed in the water then being boiled with the water. The hot water forces to break the pockets in which the oils are kept in the mangosteen pericarp and release the aromatic molecules from the mangosteen pericarp .The molecules of these volatile oils then escape from the mangosteen pericarp and evaporate into the steam. In this research, the heat loss of hydrodistillation process of mangosteen is discussed because the energy consumption is important to reduce the cost.

1.2 Problem statement

This research is to extract essential oil of mangosteen pericarp by using hydrodistillation method and analyze the chemical composition of mangosteen pericarp by using Gas chromatography- flame ionization detector analysis (GC-FID) .It is most possible that there is heat loss during the hydrodistillation process. Therefore, the heat loss analysis of the process of hydrodistillation in this research was reviewed because the energy consumption is important to reduce the cost.

1.3 Research Objectives

This research is about hydrodistillation of mangosteen pericarp but operating objective focus heat loss analysis.

1.4 Scope of Proposed Study

In order to achieve the objective, this research covers:

- i) The study of hydrodistillation of mangosteen pericarp and the chemical composition of mangosteen pericarp.
- ii) The determination of the heat loss analysis of hydrodistillation.

1.5 Significance of Proposed Study

The significance of this proposed study is to analyze the chemical composition of mangosteen pericarp by using hydrodistillation method and study heat loss analysis of hydrodistillation.

CHAPTER 2

LITERATURE REVIEW

2.1Chapter Overview

Essential oils are used in a wide variety of consumer goods such as detergents, soaps, toilet products, cosmetics, pharmaceuticals, perfumes, confectionery food products, soft drinks, distilled alcoholic beverages (hard drinks) and insecticides. An essential oil is an oil having the odour or flavour of the plant from which it comes; used in perfume and flavourings. It is a water-immiscible material produced by distillation from some plant material. Paracelsus believed every essential oil extract had a quintessence that was most effective in accomplishment of cures. The world production and consumption of essential oils and perfumes are increasing very fast. In this research the essential oil of mangosteen pericarp was isolated by hydrodistillation. The composition of the volatile oil was characterized by GC-FID.

Fundamentals theories of heat transfer such as heat loss analysis was discussed in this research. This research was focused on the heat loss analysis for hydrodistillation.

2.2Mangosteen

Mangosteen (Garcinia mangostana L.) is one of the most praised of tropical fruit. It is also known as mangostanier, mangoustanier, mangouste, mangostier (French), mangostan (Spanish), manggis, mesetor, semetah, sementah (Malaysian), manggustan, mangis, mangostan (Philippine), mongkhut (Cambodian), mangkhut (Thai), cay mang cut (Vietnamese), manggis, manggistan (Dutch), and mangostao, mangosta, mangusta (Portuguese) (Nakasone and Paull, 1998). Mangosteen (Garcinia mangostana Linn) is a tropical fruit in Guttiferae family.Mangosteen has a smooth, thick and firm rind 6-8 mm thick, pale green when immature and dark purple or red-purple when fully ripe. The edible fruit aril is white, soft, and juicy with a sweet, slightly acid taste and a pleasant aroma (Martin, 1980). It is also known as "The queen of the fruit". It is commonly cultivated in Thailand, Malaysia, and Indonesia. Mangosteen is a very slow growing and shallow-rooted tree, however, it can be productive for over 50 years.Mangosteen season in Thailand is last from May to September. Most of mangosteen is consumed

fresh or exported to foreign market. Extending shelf-life by using several food processing such as juice processing, concentrating, and drying could add value for mangosteen and create a new market. Recently, products such as mangosteen juices or dietary supplements have begun to be widespread around the world.

2.3 Importance of mangosteen pericarp essential oil in industry

Mangosteen pericarp, 6–10 mm in thickness, has been used in traditional Thai medicine for treating skin infections, wounds, and diarrhea for many years (Mahabusarakam *et al.*, 1987; Moongkarndi *et al.*, 2004). About 30% of fresh mangosteens are eaten and the rest is the peel which is not edible and becomes garbage (Sangkhapaitoon *et al.*, 2008). Zadernowski *et al.* (2007) reported that Protocatechuic acid was the major phenolic acid in the peel and rind. Several researchers recognized phenolics and anthocyanin for their antioxidant properties (Robards *et al.*, 1999; Karakaya *et al.*, 2001; Rossi *et al.*, 2003; Davalos *et al.*, 2005; Balasundram, 2006). The ethanolic fruit peel extracts from mangosteen (*Garcinia mangostana* L.) have potential for inhibiting acne - causing bacteria (Chomnawang *et al.*, 2005; Sukatta *et al.*, 2006).

2.4 Mangosteen pericarp essential oil components

Mangosteen fruit is a rich source of phenolic compounds such as xanthones, condensed tannins and anthocyanins (Fu, Loo, Chia, & Huang, 2007; Jung, Su, Keller, Mehta, & Kinghorn, 2006; Mahabusarakam *et al.*, 1987).

2.4.1 Xanthones

Xanthones have been extensively studied by various research groups (Ji, Avula, & Khan, 2007; Jung *et al.*, 2006; Mahabusarakam *et al.*, 1987). There are over 50 natural xanthones (Pedraza, *et al.*, 2008) isolated from mangosteen pericarp. Xanthones are secondary metabolites commonly occurring in a few higher plant families, and in fungi and lichens. Xanthones and tannins assure astringency to discourage infestation by insects, fungi, plant viruses, bacteria and animal predation while the fruit is immature (Akao, *et al.*, 2008). Their taxonomic importance in such families and their

pharmacological properties has aroused great interest not only for the chemosystematic investigation but also from pharmacological point of view like antimalarial (Mahabusarakam, et al., 2006; Laphookhieo, et al., 2006), antibacterial (Chomnawang, et al., 2005; Rassameemasung, et al., 2007), antifungal and antiviral properties (Gopalakrishnan, et al., 1997; Vlietinck, et al., 1998), anti-inflammatory and antiallergy (Deschamps, et al., 2007; Chen, et al., 2008), antioxidant properties (Chin, et al., 2008; Haruenkit, et al., 2007) and antitumoral properties (Nakagawa, et al., 2007; Suksamran, et al., 2006).

Several research projects show that xanthone has properties which include antioxidant activities (Suvarnakuta, 2011; Zarena and Sankar, 2009 a, b; Zarena and Sankar, 2012), analgesic and anti-inflammatory (Cui, 2010), anti cancer therapeutics (Tangpong, 2011), and Alzheimer's disease (Moongkarndi, 2010). Many scientists have reviewed the medicinal properties of *Garcinia mangostana* L. extract for antioxidant, antitumor, anti allergic, anti-inflammatory, antibacterial and antiviral activities (Caverri, 2008; Dembitsky, 2011; Kapoor, 2009). It has even been mentioned by other reviewers that mangosteen xanthones are effective for treating gastrointestinal disturbances and for wound-healing including antifungal, anti malarial, and anti-HIV (Kinghorn, 2011). This seems to suggest a potential for development as an anti-virus medicine in the future.

2.4.2 Anthocyanins

Anthocyanins are considered secondary metabolites. The purple colour of the mangosteen fruit pericarp is mainly due to anthocyanins. Among the natural food colours, the anthocyanin is the most popular colour. Du and Francis (1977) identified by thin layer chromatography (TLC), two anthocyanins in the mangosteen pericarp, cyanidin-3-sophoroside and cyanidin-3-glucoside. The major anthocyanin in mangosteen was cyanidin-3-sophoroside (Du and Francis, 1977). Anthocyanin also is a food additive E163. This natural pigment is used quite safely in food; create more attractive colours for food products.

2.4.3 Tannin

Theppoonpol (1995) reported that mangosteen peel consisted of 14.1% tannin. Tannin are polyphenolic compounds found in the plant kingdom. Tannins have molecular weights ranging from 500 to 3,000 (Viriwutthikorn, 1996). Their main characteristic is the capability of binding and precipitating proteins. Tannin can precipitate protein in wine and increase wine clarity. Tannin also increases wine astringency which will improve the taste of wine. Tannins can be used in many industries such as tanning leather, ink manufacture, particle board, cosmetics and pharmaceuticals.

2.4.4 Ferulic Acid

Ferulic acid is a naturally found phenolic acid, is a potent antioxidant able to neutralize free radicals, such as Reactive Oxygen Species (Rice-Evans et al, 1996). The syntheis of Ferulic acid was established by Dutt in 1935 when ferulic acid was used as a precursor in the manufacturing of vanillin and malonic acid. Ferulic acid possesses multiple pharmacological and biological effects including anti-inflammatory, hepatoprotective, anticancer and antioxidant properties (Anselmi *et al.*, 2004; Rukkumani *et al.*, 2004). Ferulic Acid, like many phenols exhibits antioxidant effect in response to free radicals by donating hydrogen from its phenolic hydroxyl group and also an antimicrobial agent. It is also recognized that ferulic acid exhibits a preventive effect on discoloration in various food products and a variety of physiological functions such as suppression of Alzheimer's disease, prevention of muscular fatigue, improvement in hypertension, and antitumor activity of breast, liver, and colon. Ferulic acid is also used for cosmetics, because it exhibits UV absorption effect, whitening effect and anti-inflammatory effect.

2.5 Method of extraction (hydrodistillation)

2.5.1 Introduction

Hydro-distillation is the simplest, oldest and primitive process known to man for obtaining essential oils from plants. Hydrodistillation is made with the Clevenger type apparatus. In order to isolate essential oils by hydrodistillation, the aromatic plant material is packed in a still and a sufficient quantity of water is added and brought to a boil; alternatively, live steam is injected into the plant charge. Due to the influence of hot water and steam, the essential oil is freed from the oil glands in the plant tissue. The vapor mixture of water and oil is condensed by indirect cooling with water. From the condenser, distillate flows into a separator, where oil separates automatically from the distillate water.

2.5.2 Principle of hydrodistillation

A mixture of water and mangosteen pericarp is heated to boiling. Heating causes the plant cells to rupture and the fragrance molecules to be released. The water and essential oil vapours pass through a condenser where they are cooled by cold water, causing them to condense into a liquid mixture (the distillate) which is collected. The distillate contains both water and essential oil forming two immiscible layers. Mangosteen essential oil, less dense than water, floats on the surface of the water (N.Remy, 2007).

2.5.3 Mechanisms involved in hydrodistillation

Hydrodistillation of plant material involves the hydrodiffusion, hydrolysis and decomposition by heat those main physicochemical process. Diffusion of essential oils and hot water through plant membranes is known as hydrodiffusion. When the plant material is soaked with water, exchange of vapors within the tissue is based on their permeability while in swollen condition. Membranes of plant cells are almost impermeable to volatile oils. Therefore, in the actual process, at the temperature of boiling water, a part of volatile oil dissolves in the water present within the glands, and this oil-water solution permeates, by osmosis, the swollen membranes and finally reaches the outer surface, where the oil is vaporized by passing steam. Another aspect of

hydrodiffusion is that the speed of oil vaporization is not influenced by the volatility of the oil components, but by their degree of solubility in water. Therefore, the high-boiling but more water-soluble constituents of oil in plant tissue distill before the low boiling but less water-soluble constituents. Since hydrodiffusion rates are slow, distillation of uncomminuted material takes longer time than comminuted material.

Hydrolysis in the present context is defined as a chemical reaction between water and certain constituents of essential oils. Esters are constituents of essential oils and, in the presence of water, especially at high temperatures, they tend to react with water to form acids and alcohols. Therefore, if the amount of water is large, the amounts of alcohol and acid will also be large, resulting in a decreased yield of essential oil. Furthermore, since this is a time-dependent reaction, the extent to which hydrolysis proceeds depends on the time of contact between oil and water. This is one of the disadvantages of hydro distillation.

Almost all constituents of essential oils are unstable at high temperature. To obtain the best quality oil, distillation must be done at low temperatures. The temperature in hydro distillation the operating pressure is usually atmospheric. All the previously described three effects, i.e. hydrodiffusion, hydrolysis and thermal decomposition, occur simultaneously and affect one another. The rate of diffusion usually increases with temperatures as does the solubility of essential oils in water. The same is true for the rate and extent of hydrolysis. However, it is possible to obtain better yield and quality of oils by maintaining the temperature as low as possible and thoroughly comminuting the plant material and packing it uniformly before distillation (Anonymous).

2.6 Heat loss analysis

2.6.1 Heat transfer

Heat transfer is the transmission of energy from one region to another region as a result of the temperature difference between them (Incropera, F. P. *et al.*, 2007). Heat is always transferred in the direction of decreasing temperature. Heat transfer is generally defined as having three major transfer processes: conduction, convection and radiation (Kreith and Bohn, 2001). Heat transfer plays an important role in the design of many devices, such as spacecraft, radiators, heating and air conditioning systems, refrigerators and power plants. The amount of thermal energy Q, given by

$$Q = mc\Delta T (Equation 2.1)$$

Where m is the mass (weight) of the material, c is the specific heat of the material and ΔT is the temperature difference is absorbed or liberated in a sensible heating process.

2.6.2 Heat loss calculation for round shape apparatus

The term "heat loss" commonly refers to the heat transfer of an object to its ambient environment. The energy loss of a body to the environment is mainly due to convective processes. According to the equation for heat transfer the convectively emitted energy Q increases with rising temperature differences between laboratory apparatus and surrounding medium.

$$Q = hA(\Delta T)$$
 (Equation 2.2)

Where h is for convective heat transfer coefficient, A is the area implied in the heat transfer process, ΔT is the temperature difference.

2.6.3 Glassware laboratory

Glass is an inorganic mixture of metal oxides fused together at high temperatures, which upon cooling, solidifies into the clear, rigid, noncrystalline, versatile material known widely across the globe. Glass is made chiefly from silica sand (silica, also called silicon dioxide), soda ash (sodium carbonate), and limestone (calcium carbonate). Glass is commonly used for windows, bottles, and eyewear; examples of glassy materials include soda-lime glass, borosilicate glass, acrylic glass, sugar glass, Muscovy-glass, and aluminium oxynitride. Glass plays an essential role in science and industry .Borosilicate glass, the third major group, is made mainly of silica (70-80%) and boric oxide (7-13%) with smaller amounts of the alkalis (sodium and potassium oxides) and aluminium oxide. This type of glass has relatively low alkali content and consequently has good chemical durability and thermal shock resistance (it doesn't break when changing temperature quickly). As a result it is widely used in the chemical industry, for laboratory apparatus, for ampoules and other pharmaceutical containers, for various high intensity lighting applications and as glass fibres for textile and plastic reinforcement

2.6.4 Common Efficiency of heating devices

The efficiency of an energy conversion device is a quantitative expression of this balance between energy input and energy output. It is defined as follows:

$$Device \ efficiency = \frac{Useful \ energy \ output}{Energy \ input}$$
 (Equation 2.3)

A device is a piece of equipment that serves a specific purpose. An energy conversion device converts one form of energy into another. Greater energy efficiency can reduce energy costs to consumers, enhance environmental quality, maintain and enhance our standard of living, increase our freedom and energy security, and promote a strong economy. The efficiency of the system is always lower than any one of the efficiencies of the individual components of the system. Fuel cells (Chemical/Electricity) have typical efficiencies of 20-80%.

2.7 Gas chromatography-flame ionization detector analysis (GC-FID)

Chromatography is an important analytical tool that allows for the separation of components in a gas mixture. Gas chromatography (GC) can separate volatile and semivolatile compounds with great resolution, but it cannot identify them. GC is a common type of chromatography used to separate and analyze compounds that can be vaporized without decomposition. Typical uses of GC include testing the purity of a particular substance or separating the different component and relative amounts of different components of a mixture. GC can also be used to prepare pure compounds from a mixture (Pavia et al., 2006). By calibrating GC you can find out at what time various organic compounds are being detected. Flame Ionization Detector (FID) was first developed in 1957 by scientists working for the Commonwealth Scientific and Industrial Research Organization in Melbourne, Australia (Specialty Gas Report, 2009).Flame Ionization Detector (FID) is one of the most used detectors for gas chromatography (GC). The application area is wide. For example, petrol for air planes, kerosines, are carefully analyzed with GC-FID as a routine control. It provides a high level of sensitivity combined with a wide linear range of 6 or 7 orders of magnitude (106 to 107) and limits of detection in the low pictogram. Many in the industry believe its sensitivity is so powerful it is without parallel among Gas Chromatographic (GC) detectors.

2.7.1Operation of (GC-FID) analysis

Gas chromatography is an instrumental method for the separation and identification of chemical compounds. Chromatography involves a sample (or sample extract) being dissolved in a mobile phase (which may be a gas, a liquid or a supercritical fluid). The mobile phase is then forced through an immobile, immiscible stationary phase. The phases are chosen such that components of the sample have differing solubilities in each phase. A component that is quite soluble in the stationary phase will take longer to travel through it than a component that is not very soluble in the stationary phase but very soluble in the mobile phase. As a result of these differences in mobilities, sample components will become separated from each other as they travel through the stationary phase. The diagram of the separation process is shown in Figure 2.1

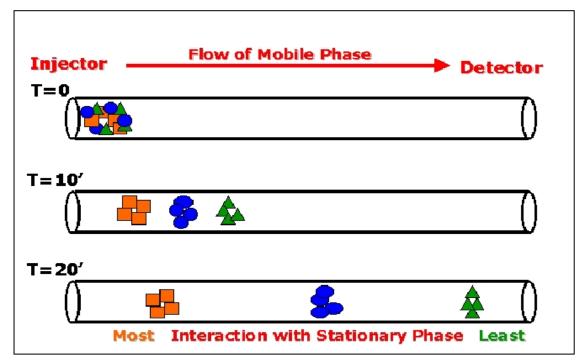


Figure2-1 The separation process

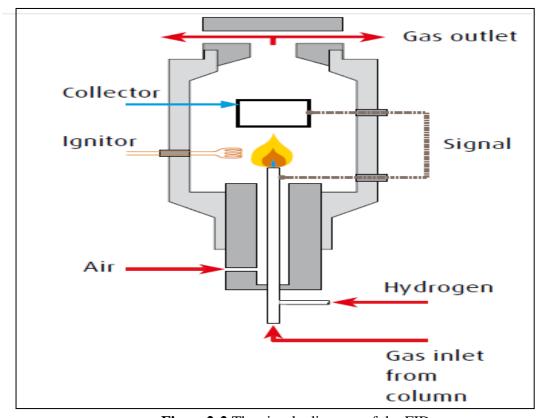


Figure2-2 The simple diagram of the FID

After the separation of the compounds, Flame Ionization Detector (FID) is used to identify each of them and determine their mass. The effluent from the column is mixed with hydrogen and air, and ignited. Most organic compounds, when pyrolysed at the temperature of a H2/air flame, produce ions and electrons that can conduct electricity through the flame. The resulting current (~10-12A) is then directed into a high impedance operational amplifier for measurement. FID exhibits a high sensitivity, low noise and is easy to use. The ionization of carbon compounds in a flame is a poorly understood process, although it is observed that the number of ions produced is roughly proportional to the number of reduced carbon atoms in the flame. The FID responds to the number of carbon atoms entering the detector per unit time. It is a mass sensitive, rather than a concentration sensitive device so this gives the advantages that changes in mobile phase flow rate do not affect the detector's response. The FID is a useful general detector for the analysis of organic compounds; it has high sensitivity, a large linear response range, and low noise. It is also robust and easy to use, but it destroys the injected sample. After detection, a signal is sent to the recording device. You will see the development of the curve on the computer screen. The time between sample injection and an analyte peak reaching a detector at the end of the column is termed the retention time (t_R). Each analyte in a sample will have a different retention time. The time taken for the mobile phase to pass through the column is called t_M . The area under the curve (automatically calculated by the computer) may be expressed in terms of concentration of the chemical compounds, by running some calibration standards at known concentration.

CHAPTER 3

METHODOLOGY

3.1 Chapter Overview

The research work focus on the heat loss of hydrodistillation process of mangosteen pericarp. To accomplish the objectives and scope of this research, the study was carried out in four stages. The first stage was sample preparation, the second stage was monitor the temperatures of round bottom flask, biomass flask and condenser part, the third stage was focused on the heat loss of hydrodistillation process of mangosteen pericarp and the final stage was studied the correlation between the concentration of ferulic acid and time. The detailed procedure of the experimental work is discussed throughout this chapter.

3.2 Overall methodology

The flow chart of overall methodology of the research was present on Figure 3.1.

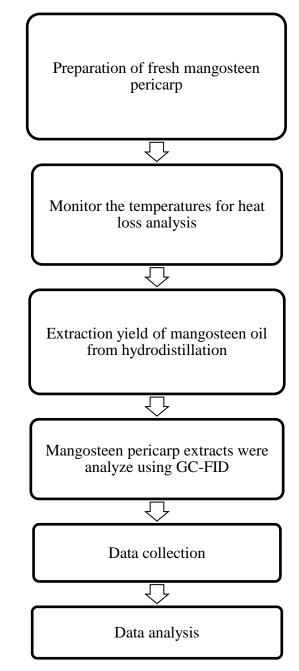


Figure 3-1 Overall methodology

3.3 Experiment methodology

3.3.1 Sample preparation

Mangosteens were bought from the fruit stall near Kuantan. After that, the mangosteen pericarps were removed from mangosteen fruits. The mangosteen pericarps were cut into smaller size and 300g of mangosteen pericarps were weighted.

3.3.2 Hydrodistillation extraction

3.3.2.1 Apparatus: Clevenger-type apparatus

The picture of the experimental apparatus used for hydrodistillation is shown in Figure 3.2. It consists of two-neck round bottom flask, biomass flask, one condenser (cooling water) and two thermometers. It also has an individual heating mantle to give heat to the hydrodistillation process. Clevenger type apparatus has its ability to capture the oil, whilst returning the excess water back into the reaction flask. The vapors produced from the mixture are passed through a long vertical glass tube (delivery tube) to a vertical condenser having a long vertical glass tube. The volatile oil is immiscible in water and, being less dense, separates out as an upper layer. A return conduit connects the base of the measuring tube to the vertical tube and allows recycling of the aqueous part of the vapors. The oil is collected at the outlet by opening a valve provided for that purpose.