PRODUCTION OF VIRGIN COCONUT OIL (VCO) VIA COMBINATION OF MICROWAVE AND CENTRIFUGATION METHOD

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ABSTRACT

In this study, the potential of the centrifugation method and the combination of microwave and centrifugation method in demulsification (emulsion breaking) of coconut milk emulsion is investigated. The conventional methods in producing Virgin Coconut Oil (VCO) by using drying, fermentation and cold pressing need longer time to break these emulsions. For the research purpose, the coconut milk without added water from local market was used as samples. For the centrifugation method, the centrifuge speeds were varied from 6000 to 12000rpm and centrifugation times were varied from 30 to 105min. For the combination of microwave and centrifugation method, the microwave powers of 360, 540 and 720Watts were used and centrifugation times were varied from 60 to 105min. From the results obtained, for centrifugation method, the highest yield of 37.3% VCO is obtained at 12000rpm and 105min; while, for the combination of microwave and centrifugation method, the highest yield of 46.88% VCO is obtained at 720W of microwave power at 12000rpm and 105min. Results showed that the combination of microwave and centrifugation method gives a better yield of VCO. Due to its fast and higher yield of VCO, combination of microwave and centrifugation method can be used as an alternative demulsification method for oil-in-water coconut milk emulsions.

ABSTRAK

Dalam kajian ini, potensi dalam pemecahan emulsi daripada emulsi santan oleh kaedah sentrifugasi dan gabuangan kaedah gelombang mikro dan kaedah sentrifugasi (pecahan emulsi) diselidiki. Kaedah konvensional dalam menghasilkan Virgin Coconut Oil (VCO) dengan menggunakan pengeringan, fermentasi dan dingin menekan memerlukan masa yang lebih lama untuk memecahkan emulsi ini. Untuk tujuan kajian, santan tanpa tambahan air dari pasaran tempatan digunakan sebagai sampel. Untuk kaedah sentrifugasi, kelajuan centrifuge bervariasi dari 6000 hingga 12000rpm dan masa sentrifugasi bervariasi dari 30 hingga 105min telah digunakan. Untuk gabungan kaedah gelombang mikro dan kaedah sentrifugasi, kuasa gelombang mikro 360, 540 dan 720 Watts dan masa sentriugasi bervariasi dari 30 hingga 105min telah digunakan. Dari hasil yang diperoleh, untuk kaedah sentrifugasi, hasil tertinggi 37.3% VCO diperoleh pada 12000rpm dan 105min, sedangkan, untuk kombinasi kaedah microwave dan sentrifugasi, hasil tertinggi 46,88% VCO diperoleh pada 720W kuasa gelombang mikro, 12000rpm dan 105min. Keputusan kajian menunjukkan bahawa gabungan kaedah gelombang mikro dan sentrifugasi memberikan hasil VCO yang lebih baik. Kerana cepat dan hasil yang lebih tinggi VCO, gabungan kaedah gelombang mikro dengan sentrifugasi boleh digunakan sebagai kaedah alternatif untuk pemecahan susu emulsi minyak-dalam-air kelapa.

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LIST OF ABBREVIATIONS

RPM	-	Revolution per Minute
EM	-	Electromagnetic
$CaCl_2$	-	Calcium Chloride
KOH	-	Potassium Hydroxide
FID	-	Flame Ionization Detector
GCMS	-	Gas Chromatography Mass Spectrometer
APCC	-	Asian Pacific Coconut Community
MSDS	-	Material Safety Data Sheet

LIST OF SYMBOLS

γ	-	Gamma
ω	-	Angular Velocity
υ_o	-	Settling Velocity
ρ_w	-	Density of Water
ρ_o	-	Density of Oil
r	-	Radius of rotation
D	-	Diameter
μ_w	-	Viscosity of Water
^0C	-	Degree Celcius
%	-	Percent
mL	-	Mililiter
rpm	-	Revolution per Minute
M	-	Molarity
cm	-	Centimeter
MHz	-	Megahertz
W	-	Watts
g	-	Gram

CHAPTER 1

INTRODUCTION

1.1 Research Background

1.1.1 Coconut Oil

Most commercial grade coconut oils are made from copra (dried kernel of coconut). It can be produced by smoke drying, sun drying of a combination of both. If standard copra is used as starting material, the unrefined coconut oil obtained is not suitable for consumption and must be purified due to unsanitary handling. The coconut oil must be further refined where the standard end product is RBD (Refined, Bleached and Deodorized) coconut oil. High heat is used in the deodorization of the oil and the oil is typically filtered through clays (bleaching) to removes impurities. Sodium hydroxide is generally used to remove free fatty acids and prolong shelf life of the coconut oil. Both chemicals and high heat are usually used to improve the yield of oil from copra. RBD process is required to make the oil clear, tasteless and odorless. This process further removes the anti-oxidant and other properties of the oil. The traditional way of producing refined coconut oil is through physical or mechanical refining (Guarte RC *et al.*, 1996).

1.1.2 Virgin Coconut Oil (VCO)

Coconut oil that is extracted from fresh mature kernel of coconut (fresh coconut kernel which is not dried) is known as Virgin Coconut Oil (VCO). Presently, Virgin Coconut Oil (VCO) is increasingly gaining wide popularity in the scientific field and among the public. It is getting global reputation as the healthiest and versatile oil and has gain a lot of attention in the world. The extraction of VCO involves a process where the oil is obtained by mechanical or natural means, without the use of heat, without undergoing chemical refining, bleaching or deodorizing which does not lead to the alteration of the nature of the oil. VCO is rich in Lauric Acid, an essential fatty acid which transforms into a compound known as Monolaurin Acid that is believed can fight viral pathogens which protects the body from bacteria, viruses and infections from parasites. Besides Lauric Acid, VCO contains a considerable amount of short-chain fatty acids such as capric, caproic and caprylic acid which were investigated to have antimicrobial and antiviral properties (Bergsson *et al*., 1998). VCO has been claimed to have numerous beneficial health effects where it lowered total cholesterol, triglycerides, and phospholipids (Delmo G. C., 2004).

Coconut oil obtained from copra, dried coconut, has no taste or fragrance, due to the refining process, whereas VCO has the natural fresh fragrance and taste of coconut which is free from rancid flavor and odors. The absence of heating and chemical treatment in the oil makes it tasty and healthy. The existing process of the production of VCO basically is conducted through oil separation from coconut milk. Coconut milk can be obtained by either pressing fresh coconut flesh without additional of water. The oil can be separated from the emulsion by means of boiling, fermentation, refrigeration or mechanical centrifuge. Separation of the oil from emulsion can also be accomplished by breaking the emulsion.

1.1.3 Uses of Virgin Coconut Oil (VCO)

There are numerous uses of Virgin Coconut Oil (VCO). VCO is one of the few herbal internally and externally supplements. VCO is known of its health benefits which it is used in heart-related problems which includes lowering of triglycerides and cholesterol level. VCO contains Lauric acid which is found in mother's milk that helps the newborn's body to fight against various diseases and helps to build their immune system. Once our body digests the Lauric acid, it is transformed into Monolaurin acid which is known for its antiviral, antibacterial, antiprotozoal, antifungal and antimicrobial properties and its action against lipid-coating of viruses. It also contains other important acids such as myristic acid, palmitic acid, caprylic acid, capric acid, stearic acid, linoleic acid, palmitoleic acid and oleic acid.

VCO is also known for its effective use in healing injuries, internally as well as externally. From ancient times, VCO is used as a protective and healing supplement for injuries. VCO can also be used as a moisturizer for all skin types, especially dry skin and aging skin, leaving skin refreshed and looking wide-awake. Moreover, it can be used as hair care product where it provides essential proteins required for nourishing damaged hair.

1.1.4 Production Methods of Virgin Coconut Oil (VCO)

One of the most widely used method in treating the oil-in-water emulsion in the production of VCO is the traditional fermentation method. This method is the natural separation of the coconut oil from water using gravity, thus no machine or any other substances are used in the extraction. First, the coconut milk is obtained from the fresh mature coconuts without adding any chemicals. The milk is then fermented in containers for approximately a day. After which, the coconut oil is then separated from the water.

This oil is then carefully filtered to obtain the pure coconut oil and what is called the Virgin Coconut Oil (VCO) (N.A. Nik Norulaini *et at.,* 2009). There are several methods used for the production of coconut oil, such as electrostatic coalescence and centrifugal methods. The existing process production of VCO basically is conducted through oil separation from coconut milk (Sukartin and Sitanggang, 2005).

In this research project, the microwave heating and separation technology is used as an alternative method for the production of coconut oil. Electromagnetic radiation in the frequency range 300MHz to 300GHz are known as microwaves, microwave energy is non-iodizing radiations that cause molecular motion by migration of ions and dipole rotations, but does not cause changes in molecular structure and wavelengths ranging from a few centimeters to a few millimeters (Abdurahman *et al*., 2006). Microwave heating offers a faster processing rate because of its volumetric heating effects. Microwave energy is delivered directly to materials through molecular interaction with the electromagnetic field. In heat transfer, energy is transferred due to its thermal gradients while microwave heating is the transfer of electromagnetic energy to thermal energy and it is more to energy conversion, rather than heat transfer. This difference in the way energy is delivered can results in many potential benefits of using microwaves for the production of virgin coconut oil. The transfer of energy does not rely on diffusion of heat from the surfaces and it is possible to achieve rapid and uniform heating of thick materials (Abdurahman *et al*., 2006).

1.1.5 Emulsion

An emulsion is a mixture consists of two immiscible liquids (usually a mixture of oil and water), are made up of dispersions of liquid in liquid as a small spherical droplets and a continuous phase. As a result of the mixing, one may obtains oil droplets in water (O/W) or water droplets in oil (W/O). The sizes of the droplets could be in the micrometer and even sub-micrometer range (D. N. Petsev, 2004). Emulsions are thermodynamically unstable due to the unfavorable contact between oil and water molecules. As a result, their physical structures will tend to change over time by various mechanisms (e.g., creaming, flocculation and coalescence), eventually leading to complete phase separation. Thus, unstable emulsions will eventually separate into two layers.

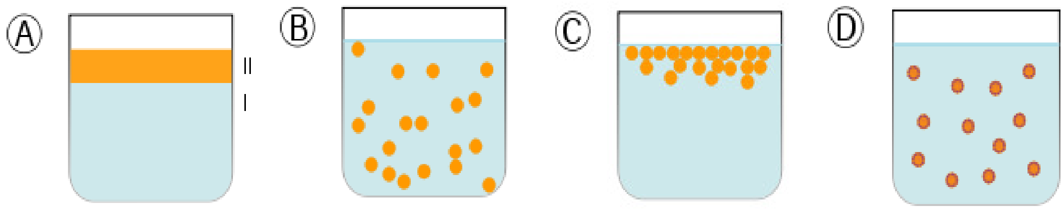

Figure 1.1: Stabilization and Destabilization of Emulsion

A. Two immiscible liquids which has not been emulsified.
B. An emulsion of Phase II dispersed in Phase I.
C. The unstable emulsion progressively separates.
D. The surfactant (purple outline around particles) positions itself on the interfaces between Phase II and Phase I, stabilizing the emulsion

Coconut milk is the natural oil-in-water emulsion extracted from the endosperm of mature coconut (Cocos nucifera L.) either with or without the addition of water. It contains fat, water, carbohydrate, protein and ash with the major components being water and fat. The emulsion is known to be naturally stabilized by coconut proteins: globulins and albumins and phospholipids. However, the coconut milk emulsion is unstable and readily separates into two distinct phases – a heavy aqueous phase and a lighter cream phase. The reason for the instability is that the protein content and quality in coconut milk is not sufficient to stabilize the fat globules (Abdurahman *et al.*,2006)

1.1.6 Separation Process

In chemistry and chemical engineering, separation process is used to transform a mixture of substances into two or more distinct products. The separated products may differ in chemical properties or physical property, such as size. Almost every element or compound is found naturally in an impure state such as a mixture of two or more substances. Most of the time the need to separate it into its individual components arises. Separation processes can essentially be termed as mass transfer processes. The classification can be based on the means of separation, mechanical or chemical. The choice of separation depends on the pros and cons of each. Usually, mechanical separations are preferred if possible due to its lower cost of operations as compared to chemical separations. Depending on the raw mixture, various processes can be employed to separate the mixtures. Many times two or more of these processes have to be used in combination to obtain the desired separation. In addition to chemical processes, mechanical processes can also be applied where it is possible.

1.1.7 Abbreviations

A list of abbreviations and concepts that need to be defined and clarified is provided below because they will be used throughout the proposal and thus it is important to avoid any misinterpretations as well as to further increase the understanding of the research report.

1 **Emulsion** is a mixture of two or more immiscible (unblendable) liquids. Emulsions are made up of a dispersed and a continuous phase; the boundary between these phases is called the interface. Emulsions tend to have a cloudy appearance because the many phase interfaces scatter light that passes through the emulsion. Emulsions are unstable and thus do not form spontaneously.

2 **Demulsification** is a process where the coconut milk emulsion is break down into liquid and oil.

3 **Microwaves** are electromagnetic waves with wavelengths ranging as long as one meter to as short as one millimeter, or equivalently, with frequencies between

300MHz and 300GHz. Microwave is used for the sending of information by radio or radar.

1.2 Problem Statement

The tradition method used in the emulsion treatment of the production of VCO is found to be limited and time-consuming. The conventional fermentation method takes longer time to separate the emulsion through the gravity. Gravity is mainly associated with the slow sedimentation process of an immiscible mixture. Sometimes, gravity separation may be too slow because of closeness of the densities of the particles and the fluid, or because of association forces holding the components together (Geankoplis, 2003). Gravity separation takes hours while microwave separation method may be accomplished in minutes or even seconds.

Moreover, the conventional methods for this process may involve the usage of chemicals and high-heating for further refining process, thus causes the loss of natural properties of the coconut oil which further affects the quality of the coconut oil. Furthermore, demand on VCO is increasing due to its health benefits. Thus, in my research, combination of microwave and centrifugation method has been discovered as an alternative method to solve these problems which it is believed can helps to save energy and time, also to improved the quality of coconut oil.

1.3 Objective

Mainly, the objective of this research is focus on the study of the potential of combination of microwave and centrifugation separation technology in demulsification (emulsion breaking) of coconut milk emulsion into water and oil (VCO). At the same time, comparison between the *Combination of microwave and centrifugation separation* *technology* and *Centrifugation method* in terms of demulsification potential is studied. Besides this, fatty acid of VCO is to be analyzed in this research.

1.4 Scope of Research

Several scopes are listed out below as to accomplish the objective of the research. The scopes of this research focus on:

i. to prove that *Combination of Microwave and Centrifugation Method* results in better yield and time saving.

ii. to prove that *Combination of Microwave and Centrifugation Method* can be an effective and alternative method to produce VCO

iii. to analyze the fatty acids in VCO

iv. to determine the temperature distributions at different locations (top, middle and bottom) for irradiated emulsions.

v. to study the effect of demusification by varying the microwave power generation.

vi. to analyze the overall potential of *Combination of Microwave and Centrifugation Demulsification Method* as an alternative energy generation for oil-in-water emulsion demulsification.

1.5 Rationale and Significance

Combination of Microwave and Centrifugation Method is introduced in this research because it is easy to operate. Beside this, time taking for to produce the coconut oil using this method is short compare. Thus, this method is much quicker compare to the traditional conventional heating method. Combination of Microwave and Centrifugation method is preferred because of its efficiency and its potential to contribute to clean products. Microwave heating technology provides convenient heating process and most of times results in better yields. Moreover, coconut oil produced using this method retains its natural scents and odors. It is environmental friendly because it does not involve any chemical addition and therefore no pollution will be created. The future for the application of microwave heating technology looks bright because of its variety of benefits.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Coconut oil contributes about 10% of the total oils and fats in the world market. Several methods are currently practiced in the production of coconut oil either from fresh coconut meat or copra (dried coconut kernel). These technologies include the (i) wet process, (ii) dry process, and (iii) solvent extraction.

The recovery of coconut oil by the traditional wet process is usually low, which is about 30% (Thieme *et al.,* 1968). Furthermore, the oil obtained is of poor quality due to the high moisture content (MC), dark color, and short shelf life (Hagenmaier *et at.,* 1973). The process is also energy- and time-consuming.

2.1.1 Wet Process

The wet process is a process where the coconut meat is grinded with water and is then filtered to produce coconut milk or coconut cream (oil-in-water emulsion). This emulsion contains protein and coconut oil which can be separated either through common kitchen utensils or hydraulic presses. Using this process, the coconut oil is extracted from the coconut meat without drying it first. However, coconut oil extraction by these wet process techniques has not been commercially successful.

2.1.2 Dry Process

The present commercial technique for coconut oil production is through the dry process. Fresh coconut meat is dried first which is then used to press out the oil where the copra is first cleaned, ground, steamed, and pressed through an expeller for coconut oil extraction. This extracted oil is then further purified by neutralization, bleaching, and deodorization to remove free fatty acids, odors, flavors and pigments.

Coconut oil has been exclusively marketed using the traditional fermentation method. This method is the natural separation of the coconut oil from water using gravity, no machine or other substances are used in the extraction and therefore it is time-consuming. First, the coconut milk is obtained from the fresh coconuts without adding any chemicals. The milk is then fermented in containers for approximately half a day. After which, the water is separated from the oil where the lighter coconut solids (curds) float to the top while the heavier water sink to the bottom. The coconut oil is in between the curds and the water. This oil is then carefully filtered and separated. The oil is then slightly heated for a short time to drive off excess moisture and produce a more purified product and to extend shelf life. Here, produce the VCO which retains its natural scent and taste of coconuts. This fermentation method is a traditional method of VCO production that has been used for hundreds of years.

2.1.3 Solvent Extraction

Solvent extraction is a process where an appropriate solvent, such as benzene or n-hexane is used. Through this process, even though the oil recovery is high, it is rarely applied due to its high risk and high investment cost (Y.B. Che Man *et al.,* 1997).

2.2 Microwave Heating Technology

Microwave Heating Technology uses electromagnetic waves that pass through material and cause its molecules to oscillate. Thus, heat is generated. Microwaves form a part of the electromagnetic spectrum with the wavelength ranging from 1cm to 1m. In order to avoid interference with radar and telecommunication activities, which also operate in this ranging, most commercial and domestic microwave ovens operate at 2450 MHz, or equivalently 12.25cm. The difference between microwave energy and other forms of radiation, such as X-rays and γ-rays, is that microwave energy is non-ionizing and therefore does not alter the molecular structure of the compounds being heated, it provides only thermal activation.

Microwave heating has been found to be a very convenient thermal source not only in the kitchen but also in a chemical laboratory. There is the possibility of the application of a conventional microwave oven to carry out chemical reactions. It has been found that many reactions progress much faster upon microwave irradiation than with traditional heating techniques. In conventional thermal heating, energy is transferred to the material through convection, conduction and radiation of heat from the surfaces of the material whereas, in contrast, microwave energy is delivered directly to the material through molecular interaction with the electromagnetic field. In heat transfer, energy is transferred due to thermal gradients, but microwave heating is a process where the electromagnetic energy is transferred to thermal energy, which is energy conversion rather than heat transfer (Thostenson and Chou, 1999). This difference in the way energy is delivered results in many potential advantages of applying microwave heating technology due to its feature of penetrating power where it distributes energy within the bulk of most materials, rather than just on the surface of materials. The delivering of energy does not rely on diffusion of heat from the surfaces and it is possible to heat materials rapidly and uniformly.

The application of microwave irradiation to activate and accelerate organic reactions has taken a new dimension and has experienced exponential growth in the last eight years. Microwave irradiation is becoming increasingly popular both in industry and in academic. The future for the application of microwave technology looks bright because of its efficiency and its potential to contribute to clean products (Rashmi Sanghi, 2000).

Microwave irradiation is chosen as a method for demulsification due to its remarkable decrease in time and improved yield of products. Furthermore, it offers a clean, inexpensive and convenient heating process. It is believed that microwave heating technology can be developed which it simplify time consuming conventional process, increase overall efficiency of the processes and reduce pollution of the environment.

2.2.1 Study on Demulsification of Water-in-Oil Emulsions via Microwave Heating Technology

In this study, a batch microwave heating process of 2450 MHz was examined on crude oil emulsions. The mechanism of microwave heating is essentially that of dielectric heating. After exposing the emulsion to the microwave electromagnetic EM field, molecular rotation and ionic conduction due to the penetration of EM into the emulsion are responsible for the internal heating. In this study, microwave demulsification method was applied on water-in-oil emulsions with exposure time varied from 20 to 180 sec. Transient temperature profiles of water-in-oil emulsions inside a cylindrical container (top, middle, bottom) were measured. From the results obtained, the temperature rise at a given location was almost linear. The rate of temperature increase of emulsions decreased at higher temperature due to decreasing dielectric loss of water. Results of this work show that microwave radiation is a dielectric heating technique with the unique characteristic of fast, volumetric and selective heating is appropriate and has the potential to be used as an alternative way in the demulsification process and it does not require chemical additions (Abdurahman *et al*. 2006).

2.2.2 A Continuous Microwave Heating of Water-in-Oil Emulsions: An Experimental Study

In this study, a microwave demulsification method was utilized in a 50-50% and 20-80% of water-in-oil emulsions with varied microwave exposure time. Temperature profiles of water-in-oil emulsions inside a cylindrical container (top, middle, bottom) were measured. The temperature rise at a given location was linear. The rate of temperature increase of emulsions decreased at higher temperature due to decreasing dielectric loss of water. Due to its fast, volumetric and selective heating, microwave heating can be used as an alternative demulsification method for water-in-oil emulsions (Abdurahman *et al*. 2006).

This study showed that microwave radiation can be an effective tool to separate water from dispersed water-in-oil emulsions. Microwave heating provides a new option in breaking water-in-oil emulsions and enhances gravity sedimentation to separate the emulsions into water and oil layers. Thus, it is believed that microwave radiation can also be effective in separation of coconut oil from oil-in-water emulsions.

2.2.3 Microwave Heating and Separation of Water-in-Oil Emulsions: An Experimental Study

In this study, microwave demulsification method was applied in a 50-50% and 20-80% water-in-oil emulsions with microwave exposure time varied from 20-180 sec. Transient temperature profiles of water-in-oil emulsions inside cylindrical container (top, middle, bottom) were measured. The temperature rise at a given location was almost horizontal (linear). The results showed that the average rates of temperature increase of

50-50% and 20-80% water-in-oil emulsions are 0.351 and 0.437^{o}C sec^{-1}, respectively. The rate of temperature increase of emulsions decreased at higher temperature due to decreasing dielectric loss of water. These results indicate that microwave demulsification of water-in-oil emulsions does not require chemical additions and has the potential to be used as an alternative way in the demulsification process (Abdurahman *et al*., 2010).

In this study, from the temperature distribution profiles of irradiated emulsion, it showed that water-in-oil emulsion has been heated quickly and uniformly by microwave rather than by conventional heating. Microwave radiation appears to provide faster separation and thus it is chose to be one of the methods for my research.

2.2.4 Effect of Salinity, Temperature, Water Content, and pH on the Microwave Demulsification of Crude Oil Emulsions

The main objective of this work is to investigate the effect of a set of crude oil emulsion variables, including pH and salt and water contents, upon the microwave demulsification process. A series of batch demulsification runs were carried out to evaluate the final emulsified water content of emulsion samples after the exposure to microwaves. Tests were performed at distinct heating temperatures, using water-in-heavy crude oil emulsion samples containing different salt and water contents and pH. Well-defined temperature programs were established to control the amount of energy applied to the emulsion and, ultimately, the viscosity. Higher microwave demulsification efficiencies were achieved for emulsions containing high water contents, except when high pH and salt contents were simultaneously involved (Montserrat Fortuny *et al*., 2006).

2.2.5 Separation of Water-in-Oil Emulsions by Freeze/Thaw Method and Microwave Radiation

In this journal, the demulsification has been achieved with the use of Freeze/Thaw (F/T) method and microwave radiation method. The object of investigation is emulsion samples prepared by mixing the metal-working-oil, FESOL 09, produced by FAM, Krusevac, Serbia, and de-ionized water. F/T method has been successfully applied for the removal of oil from emulsions in our previous work. In this work, microwave radiation method has been additionally used for separation and enhanced heating of emulsion samples. The efficiency of oil removal has been improved with the assistance of microwave radiation method, up to 90% (Valdana Rajakovic and Dejan Skala, 2005).

From my study of the journals stated above, it can be concluded that microwave radiation is a heating technique with the unique characteristics of fast, volumetric and effective heating is feasible. Furthermore, this technology does not require chemical addition and it provide faster separation than the conventional heating method. Thus, microwave heating method is chosen to be applied in my research where the coconut milk (oil-in-water emulsion) is used to obtained Virgin Coconut Oil (VCO). The objective of my experimental work is to prove that microwave heating method has the potential in demulsification of coconut milk (oil-in-water emulsion) and it can be an alternative way in the demulsification process.

2.3 Centrifugal Separation Processes

In centrifugal separations, the particles are separated from the fluid by centrifugal forces acting on the particles of various sizes and densities. Two general types of separation processes are used: centrifugal settling and sedimentation.

Use of centrifuges increases the forces on particles many fold. Hence, particles that will not settle readily or at all in gravity settlers can often be separated from fluids