Soxhlet Extraction of Oil From *Aleurites Moluccana* Nut : The Effects of Extraction Temperature and Solid/Liquid Ratio

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***Abstract*—The world-wide consumption of vegetable oils to increase by at least 50% for the past decade. Due to this fact, various plants were studied for their potential sources of vegetable oil. For this study, *Aleurites Moluccana* oil was extracted using Soxhlet method and the effect of extraction temperature and solid to liquid ratio were analyzed. From the results obtained, candlenut yield quite high percentage oil and showed its potential as an alternative sources of vegetable oils. For Soxhlet extraction of candlenut oil, the oil yield percentage increases as temperature and solid to liquid ratio increases. By combining these two parameters, the optimum temperature and solid to liquid ratio were 95°C and 1g/45 mL respectively with yield of 45.17%. From the GCMS analysis, the major constituents of candlenut oil found were linoleic acid, palmitic acid, oleic acid and stearic acid.**

***Keywords*— *Aleurites Moluccana,* Soxhlet extraction, temperature, solid/liquid ratio**

I. INTRODUCTION

Vegetable oil is best defined as a triglyceride, an ester which is derived from glycerol and three fatty acids extracted from a plant [1]. They can be extracted from different part of plant such as seeds (including sunflower, safflower, and cotton), nuts (including peanut, soybean, almond and walnut)or kernel. The unique chemical structure of vegetable oils with unsaturation sites, epoxies, hydroxyls, ester and other functional group enable them to undergovarious chemical transformation [2]. Due to this fact, they are widely used in various industrial applications such as in the production of plastics, solvents, resins, plasticizers and surfactants. In addition, they also have poor viscosity index, low temperature properties and low oxidation stability [3-6].

Due to the high demand of fossil feedstock, the usage of renewable resources for the preparation of new materials is an alternative option for reducing it. Vegetable oils are potential bioresources that are renewable and abundantly available [7]. Not only that, the multipurpose functions of vegetable oils also leads to the usage of vegetable oils in various industries especially in biodiesel, food, pharmaceutical and cosmetics industries.This leads to the world-wide consumption of vegetable oils to increase by at least 50% for the past decade [1,8]. Nowadays, various new plants discovered as potential producer of vegetable oils to meet the world demand of vegetabl eoils. Various method have to be studied to explore the best technique to extract the vegetable oils at maximum yield.

Aleurites molucanna or candlenut, is a flowering tree that belongs to *Euphorbiacae* family. The name originates from the fact that the nutscan be burn with smoking flame. Candlenut trees are considered as native plants in most countries in South East of Asia or also known as Indo-Malaysia areas such as Malaysia, Polynesia, Indonesia, Brunei, Cambodia and China.In ancient times, they were introduced to Pacific islands district, and they can be primarily found in cultivation in main islands in Hawai nowadays [9]. Every parts of candlenut trees can be used for many purposes. In Polynesia, the seeds and oil are burned for illumination, thus give its local name as 'kukui' which means light by the natives. Other than that, traditional medicine were made from their seeds, leaves, flower and bark to treat various type of illnesses including fever, headache, tumors, diarrhea and asthma. However, candlenut found to be toxic, thus making it not suitable for consumption. Various parts of candlenut tree also extracted as dye to colour tapa cloth, canoes and even for tattooing [9-11]. The leaves shows an anti-inflammatory activity thus revealing its potential as a cure that can reduce swelling or inflammatory [12]. In addition, the methanolic extracts of the dried leaves also show antipyretic and anti-inflammatory properties due to presence of alkaloids, sterols and flavonoids [13]. Other than that, the presence of *n*-hentriacontane, phytosterols and triterpenes in hexane extracts of candlenut leaves have shown ananalgesic properties [10]. Candlenut waste can be used as a potential adsorbent in the removal of cationic dyes methylene blue (MB),rhodamine B (RhB),as well a cationic dyes in wastewater [14].

One of the earliest technique discovered to extract vegetable oils is pressing method, which can be classified into two; cold press and hot press. The only difference between these methods isthe seeds are being preheated for hot press and no thermal treatment involved for cold press. The oil yield obtained by cold press is lower compared by hot press. As a consequence, a pretreatment is required to increase the oil yield [15]. Mechanical press also gives high oil yield, nearly 65%. However, they need a further separation process such as filtering and degumming. In addition, this technique is not as efficient to extract some non-edible oil and for different types of seed[8]. One of examples of pressing method is hydraulic press which uses hydrostatic pressure transfer theory and liquid as the pressure transmission medium, to squeeze the grease out of oilseed. However, this technique required high labor cost and time consuming, which make this technique is not economical [16]. Next, screw press that uses screw axis to force the feeding process, through the processes of crushing, squeezing and finally oil extraction. This drawbacks of this technique are time consuming and costly since that they undergo various processes before the extraction [16].

Soxhlet extraction technique is a solvent extraction method that requires specific apparatus called the Soxhlet apparatus. In Soxhlet extraction technique, finely grounded samples are placed in a porous bag made of strong filter paper or cellulose located in the thimble.

Suitable solvent was heated at the bottom flask and vaporizes into the thimble, condenses in the condenser and drips down into the bottom flask. This technique is suitable for small scale setting. This technique is cost effective, give high recovery and can process a large sample sizes [16].

For current study, *Aleurites Moluccana* oil was extracted using Soxhlet method and the effect of extraction temperature and solid to liquid (S/L) ratio were analyzed.

II. METHODOLOGY

A. Sample Preparation

Candlenuts were purchased from a hypermarket located in Shah Alam, Selangor. The nuts were washed thoroughly to remove any dirt on the nuts. The nuts were grinded to smaller size and were left in an oven for 4 hours at 105°C to remove any moisture content that may affect the extraction products. Before extraction method, the nuts will be grinded using Waring blender until they are fine enough to be placed in the thimble of the Soxhlet extractor. The grounded nuts were passed through sieve shaker to obtain particles of 710 µm in diameter [17].

B. Soxhlet Extraction of Candlenut Oil

Methanol was used as the solvent. 5 g of fine candlenut was placed in the thimble of the Soxhlet apparatus. 150 mL of methanol was placed in the round bottom flask and attached to the Soxhlet extractor. The temperature was set at 75°C as boiling point for methanol was 64.7°C and the extraction time was set at 3.5 hours. After the extraction process finishes, the collected samples were centrifuged to ensure no solid particles existed in the oil at 500 rpm for 5 minutes. Then, the samples were placed in a rotary evaporator to remove any solvent left in the extracted oil. The mass of oil was weighed to calculate the percentage of oil yield. The oil was stored in cold and dark place prior to analysis [17]. The experiment was repeated at different temperature of which were at 80 °C, 85 °C, 90 °C and 95 °C and different volume of solvent to varies the (S/L) ratio at 1:25, 1:30, 1:35, 1:40, 1:45 and 1:50. To calculate the yield of candlenut oil, the following equation was used [17] ;

$$Oil\ yield == \frac{Mass\ of\ oil\ after\ extraction}{Mass\ of\ candlenut\ used} x\ 100\ \%$$

C. GCMS Analysis

The extracted candlenut oil was analyzed using Gas Chromatography Mass Spectrometry (GC/MS) to identify the chemical composition of the oil. Electron impact-MS of the extracted components were performed at electron energy of 70eV with a source temperature of 200°C, and a scan range of 20-550 amu with a rate of 0.81 scan per second. The column used was a 30 m x 0.25 mm x 0.25 µm cross-linked methyl siloxane fused silica capillary column at 100°C for 5 minutes, then 10°C for 1 minute to 270°C for 40 minutes. Helium was used as a carrier gas at flow rate of 1 mL /min. The split less injector was kept at 250°C. Peaks of special interest (saturated and unsaturated fatty acid) were reconfirmed by comparison to the retention time and spectra of the authentic standards with the gas chromatography [18].

III. RESULTS AND DISCUSSION

A. The Effects of Temperature

The moisture content of candlenut was found to be at 3.18%. For this study, candlenut was extracted using Soxhlet method.

To study the effect of temperature to the candlenut oil yield, the lab experiment was done at different temperature of 75°C, 80°C, 85°C, 90°C and 95°C. Figure 1 shows the effect different extraction temperature on candlenut oil yield.

From Figure 1, the optimum temperature for extracting candlenut oil was 95°C with oil yield of 33.55%. In addition, the graph clearly shown that as the temperature increases, the oil yield percentage also increases. The oil yield rose rapidly with temperature up to 80°C (21.36%) nearly doubled from the percentage at 75°C (10.52%). Then, the percentage continued to increases as extraction temperature increases until it reached highest optimum percentage of oil yield (33.33%) at temperature of 95°C.

The trend obtained was similar with report by Nwanabbe (2012) that study the kinetics and thermodynamics study of oil extraction from fluted pumpkin seed using the same Soxhlet method. From his study, the oil yield percentage increases as extraction temperature increases. In addition, according to (Adeeko and Ajibola, 1990), the increase in oil yield percentage is due to a void resulting from rupturing of oil cell walls, thus creating a migratory space for the content of the oil bearing cells [19]. In addition, that the increase in temperature lowers the viscosity of the oil, releases oil from the intact cells and draws out the moisture, thus increasing the oil yield [20]. Furthermore, increase of extraction temperature results in an increase in diffusion rates, allow better penetration into the pores and between the matrix particles, thus improved mass transfer and last, weakening and distruption of strong interactions between analytes and matrix components [21].

Through preliminary results, oil yielded at temperature higher than 100°C became darker in colour compared than other oil that yielded at temperature lower than 100°C. The boiling point for methanol was 64.7°C, thus higher temperature could results in possible degradation of phenolic compounds, caused by hydrolysis, internal redox reactions and polymerization [20, 22]. To preserve the chemical composition of the candlenut oil, the extraction was done at temperature below than 100°C.

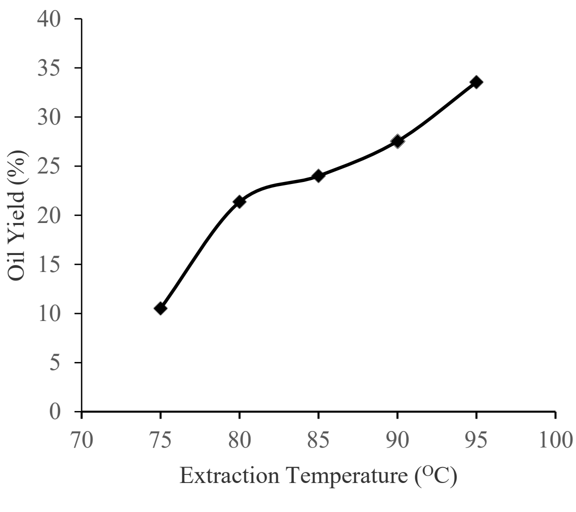

Figure 1: Effect of temperature on candlenut oil yield

B. The Effects of Solid to Liquid Ratio

For this study, the effect of S/L ratio to percentage of candlenut oil yield was examined by varying the S/L ratio from 1 g/30 mL to 1 g/50 mL. Figure 2 showed the effect of solid/liquid ratio to percentage of candlenut oil yield.

From Figure 2 below, the optimum solid to liquid ratio for candlenut oil yield was 1g/45mL with oil yield of 45.17%. In addition, the trend shows that the candlenut oil yield percentage increase steadily as the S/L ratio increase from 1g/25mL (33.55%) to 1g/45mL (45.17%). There is no significant difference when the ratio increased to 1g/50mL. The trend obtained was similar with trend obtained by Meziance and Kadi (2008) that studied the kinetics and thermodynamics of oil extraction from olive cake. They reported that as volume of solvent increases, the concentration of driving force increases [23]. Nwannabe (2012) also obtained the same trend for his study and reported that the increment in oil yield percentage also was a result of increased washing of the oil extracted away from the particle surface by the solvent as the volume of solvent increases [20].

In addition, at the initial extraction rate, the oil concentration was low in the solvent and mass transfer effect causes the oil to diffuse quickly from the candlenut to the solvent. When the maximum amount of extractable oil was reached, the oil yield remains the same even after increasing the S/L ratio as 225 mL of methanol is sufficient to bring the oil solute to equilibrium. Furhtermore, Zhang et al. (2007) explained in his studies that the chances of bio-active components to come into contact with extracting solvent increased when the amount of solvent increased, thus higher leaching-out rates leads to the increment of oil yield [24].

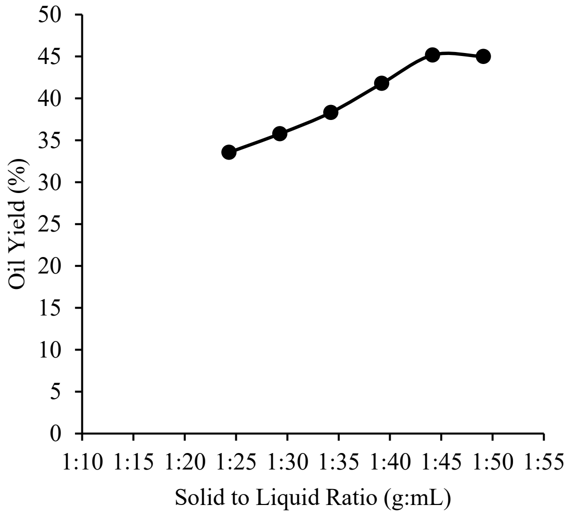

Figure 2: Effect of solid to liquid ratio on candlenut oil yield

C. GCMS Analysis

From Table 1, the major constituents of candlenut oil found were linoleic acid (9,12-Octadecadienoic acid), palmitic acid (Hexadecanoic acid), oleic acid (9-Octadecenoic acid) and stearic acid (Octadecanoic acid). The fatty acid can be classified as unsaturated fatty acid (oleic acid and linoleic acid) and saturated fatty acid (palmitic acid and stearic acid). In comparison, Norulaini et al., (2004) reported that GC analysis of candlenut seeds oil contain mainly stearic acid (54%), linoleic acid (20%) and oleic acid (16%).

From Table 1, the GCMS analysis showed Hexadecanoic acid (palmitic acid) at retention time of 32 min, 9,12-Octadecadienoic acid (linoleic acid) at 38.97 min, 9-Octadecenoic acid (oleic acid) at 39.98 min and Octadecanoic acid (stearic acid) at 40.60 min. Oils with higher amount of oleic, linoleic and linolenic acids are most likely used in the edible vegetable oil industry or in pharmaceutical industries as consumable sources. However, candlenut found to be toxic, thus making it not suitable for consumption [25].

In addition, oil with high content of lauric acid are most likely used in soap making or cosmetic products. This is supported by that stated that candlenut oil are being used in cosmetics preparation for cleansing as they dries faster than other oil, for example, linseed oil. Other than that, they are also being used in soap making and as the ingredients in preservative, varnishes and paint [26].

Linoleic acid is one of the essential fatty acid polyunsaturated that is needed by human body, other than linolenic acid. Higher intake of linoleic and linolenic acid increases HDL-cholesterol and decreases LDL-cholesterol. In contrast, high intake of oleic acid decreases LDL-cholesterol without affecting HDL-cholesterol level. Ailments such as loss of hair, liver degenaration, kidney degenaration, excessive sweating accompanied by thirst, drying up of gland, behavioural disturbance and eczema-like skin eruptions are example of effects that may affect human body due to insufficient of linoleic acid in the body [25, 26].

Table 1 : Chemical constituent of candlenut oil

No	Retention Time (min)	Chemical Constituent
1	7.92	9-Hexadecenoic acid
2	13.21	Estragole
3	31.58	Hexadecanoic acid
4	31.80	Tridecanoic acid
5	31.83	Isopropyl Myristate
6	32.00	**Hexadecanoic acid**
7	32.08	Pentadecanoic acid
8	32.32	Dodecanoic acid
9	38.97	**9,12-Octadecadienoic acid**
10	39.93	11-Octadecenoic acid
11	39.98	**9-Octadecenoic acid**
12	40.60	**Octadecanoic acid**

IV. CONCLUSION

From the results obtained, candlenut yield quite high percentage oil and showed its potential as an alternative sources of vegetable oils. For Soxhlet extraction of candlenut oil, the oil yield percentage increases as temperature and solid to liquid ratio increases. By combining these two parameters, the optimum temperature and solid to liquid ratio were 95^{o}C and 1g/45 mL respectively with yield of 45.17%. The major constituents of candlenut oil found were linoleic acid, palmitic acid, oleic acid and stearic acid.

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