Soxhlet Extraction of oil from Candlenut (*Aleurites Moluccana*): Effect of Solvent and Extraction Time

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***Abstract*—Candlenut oil was extracted using soxhlet extraction technique. The types of solvent and extraction time were optimized using one-factor-at-a-time method. The ground candlenut samples were extracted using ethyl acetate as a solvent. The optimum time for the extraction was found to be 3.5 hours with a highest yield of 72.33%. The highest yield was produced by using ethyl acetate, 72.33%, followed by acetone 46.65%, ethanol 10.1% and the least yield produced by methanol 3.41%. The extracted candlenut oil was analyzed with GC-MS for its chemical constituents were tested with GC-MS analysis and it contains palmitic acid and oleic acid.**

***Keywords*— Aleurites Moluccana, Extraction time, GC-MS, Solvent, Soxhlet extraction**

I. INTRODUCTION

Vegetable oils are made up of ester compounds and they contain glycerol molecule associated with three saturated or unsaturated fatty acids [1]. They are primarily consists of triacylglycerol with 95 - 98% and other complex mixtures of minor compounds with composition of 2- 5%. These compounds are from a broad range of chemical nature [2]. Majority of vegetable oils are recovered from beans or seeds. They normally provide two valuable commodities which are fatty oil and a protein-rich meal [3]. Variance in the distribution and composition of the vegetable oil are due to the agronomic and climatic conditions, trait of the seed and fruit, oil extraction system and refining operations [2].

Vegetable oils are potential bio resources that are renewable and abundantly available [1].Traditional vegetable oils such as linseed, cotton seed, soybean, sunflower, castor, peanut, coconut and palm have been used as the main alternative of fuels applied in diesel engines [4]. They have a heat capacity of approximately 90% of that of diesel fuel [5]. Degummed sunflower oil has tested as a fuel on an extensive range of diesel-engine agricultural tractors. The performance of all the tested tractors was very identical to that with diesel fuel. Long-term activity of direct injection diesel engines on sunflower oil shows a result of carbon build-up on injector nozzles, sticking piston rings and lubricating oil polymerization. Apart from that, as a substitute for diesel fuel, transesterification of sunflower oil aid by alcohol and a catalyst also produced a fuel. Long-term activity of a direct injection diesel engine on ester fuel turn out exceptionally good [6].

Candlenut (*Aleurites moluccana*) is a common culinary ingredient in Southeast Asian region, where it is widely used in food and have medicinal value since ancient times. Depending on the region, it is also known as kemiri, Indian walnut, candleberry, buah keras (in Malaysia) or kukui nut. [7]. Candlenut is a medium-sized tree with the height up to 20 meter tall. It has wide spreading or pendulous branches. The bark is greyish-brown in color with small vertical lines [8]. The oil content of the candle nut is high which is about 60% of the composition and it also contains almost 39.3% unsaturated fatty acids which contributes to its great nutritional value [9]. It also has been suggested that the existence of some chemical components make this oil applicable effectively for the treatment of skin problems such as eczema, psoriasis, chemotherapy and radiotherapy burn [10]. The oil extracted from candlenut contains high content of unsaturated fatty acid including oleic acid and linoleic acid which makes it possible alternative oil for human consumption and also for cosmetic application. The demands for vegetable oil sources keep growing and candlenut can be categorized as potential alternative source of vegetable oil with a versatile application in various industries [7].

Cold pressing extraction technique is generally favoured in the production of high–quality oil. Oil extraction by cold pressing grant important advantages in sustaining majority of the bioactive compounds such as flavonoids, necessary fatty acids, phenolics and tocopherol in the oils. The disadvantage of this technique is that it gives a low oil yield [11]. Cold–pressed oils are treated as healthy oils that are significant to human nutrition. This is due to the favorable polyunsatu-rated fatty acid content, notably linolenic acid and linoleic acid present in the oil [12]. Studies had been done previously on extraction of fundamental oil from Citrus fruits by using cold pressing technique. The extraction duration of 60 minutes produce 0.05% yield of oil extract [13].

Soxhlet extraction technique has been used frequently for a very long time. This conflict is bolstered by the way that soxhlet has been a typical strategy amid over one century and, at present, it is the essential reference to which the execution of other extraction technique is thought about [14]. The most critical preferences of traditional soxhlet are that the specimen is much of the time conveyed into contact with the fresh fragmant of the dissolvable. Along these lines, this uproots the transfer equilibrium. The temperature of the framework remains high since the heat acquainted with the refining flask achieves the extraction pit to some degree. No filtration is needed after the extraction process. One of the way to increase the sample throughput is by introducing concurrent extraction in parallel. This is done since the essential hardware is modest. It is an extremely straightforward methodology which needs minimal specific preparing. It has the prospect to extract more specimen lump compared to most of the present techniques such as microwaveextraction, supercritical fuids extraction and many more [14].

II. METHODOLOGY

A. Materials and sample preparation

Candlenuts were purchased from a local grocery store. They were cleaned and stored at ambient temperature. The dust and debris were removed. The cleaned seeds were ground using a dry mill and sieved through a sieve shaker (pore size 700µm).. Ethyl acetate, methanol, ethanol and acetone were used as a solvent.

B. Soxhlet extraction of candlenut oil

Oils were extracted from candlenut by the process of soxhlet extraction. Five grams of coarsely ground candlenut were weighed and transferred into a extraction thimble. They were then placed in the soxhlet extractor apparatus fitted with a condenser, which was placed on a 500-mL distillation flask containing 150 mL of ethyl acetate. Candlenut were then extracted under reflux with the solvent for time period of 2 hours [15].

Soxhlet Extractor with round-bottom flask was used. The cylindrical Soxhlet extractor was put onto the round-base flask containing the extraction solvent. A bulb-condenser was equipped at the top of the soxhlet, operated on cooling water. The solvent was heated to reflux. The solvent vapour moves along a distillation arm and surges into the chamber lodging the thimble pressed with solid seeds. The condenser ensures a zero loss of any dissolvable vapor. The vapor cools and dribbles once again into the chamber. At the point when the Soxhlet chamber is full, the chamber is naturally exhausted through a siphoning activity along a unique side arm, with the solvent running back, down into flask. This cycle may be allowed to repeat many times until extraction is complete [16]. The extraction were then repeated for time period of 2.5 , 3, 3.5 and 4 hours.

The optimum extraction time was then kept constant to evaluate the effect of solvent on the candlenut oil extraction. Three different solvent were tested which were methanol, ethanol and acetone. The temperature of the soxhlet apparatus was kept slightly above the corresponding boiling point of each solvent. This was done to eliminate error that may arise from the efficiency of the apparatus. Solid to liquid ratio was kept constant for every run at 1:30 which is 5g of solid to 150ml of solvent.

The oil samples were then centrifuged in order to remove any solid particles from the samples and were treated with rotary evaporator to expel the solvent from the extract. The connected vacuum pump with the rotary evaporator is utilized to expel the solvent totally.

$$\text{Oil yield (\%)} = \frac{\text{weight of oil obtained after extraction}}{\text{weight of Candlenut (dry materials)}} \times 100\ \%$$

C. GC-MS Analysis

The sample was the analyzed using GC-MS. The composition of the fatty acid in the oil is then analyzed by using GC/MS analysis. The equipment is controlled by computer; this also acquires the data and is used in its processing. This mix is exceptionally flexible as the gas chromatograph has the ability to isolate extremely complex blends of organics while the mass spectrometer shells each compound eluting off the GC section with electrons i.e. a high electrical potential (ordinarily 50-70 eV) and crushes every atom to ionic sections. A magnetic separator detects and separates the ionised molecular fragments according to their mass and charge [17]. The conditions used were an initial temperature of 50 °C, which was then increased to 80 °C at 10 C min−1, before increasing to 180 °C at 5 °C min−1 and up to 250 °C at 15 °C min−1. This temperature was then maintained for 5 min. The temperatures of the injector and detector were 280 and 310 °C, respectively [18]

RESULTS AND DISCUSSION

A. The effects of different extraction time

Extraction time one of the process parameter that would influence the extraction yield of candlenut oil. The effect of different extraction time was studied by using ethyl acetate as a solvent. Figure 1 shows the effect of extraction time on oil yield. It can be seen that the oil yield increased gradually with extraction time from 2 hours until the maximum yield obtained from 3.5 hours of extraction. The optimum oil yield of 72.33% yield was obtained at 3.5 hour However, a sharp decrease in the oil yield was observed beyond 3.5 hours.. Beyond this period, the yield decreased rapidly to 46.05% which was also the lowest yield obtained.

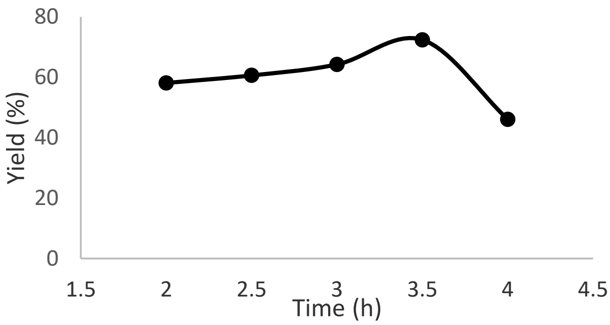

Fig 4.1: Effect of different time of extraction on candlenut (*Aleurites moluccana*) oil yield. (Conditions: ethyl acetate as a solvent, extraction temperature 80°C, solid-to-liquid ratio 1:30 g/mL)

The same trend was produced by the experiment conducted by Mogire (2013), which proves that the yield increase with extraction time up to certain point. Ma et al., (1998) studied the effect of reaction time on transesterification of beef tallow with methanol also recorded the similar trend. The extraction behaviour of ethyl acetate after optimum extraction period could be attributed to the possible formation of volatile degradation products which may have caused the decrease in the amount of oil extracted [19].

Previous study conducted by Aida et. Al., (2011) reported that prolonged extraction would prompt a decline in the phenolic content of crude extract. This is due to the oxidation of phenolic compounds that was possible to be occurred by increasing the exposure to environment factors such as light and oxygen [20]. These phenomena could be well explained by Fick's second law of diffusion, which indicates that after a certain period, there will be a final equilibrium between the solute in the solid matrix and in the bulk solution [20].

B. Effect of different types of solvent

The extraction of oil from candlenut seeds was performed using different solvents including acetone, methanol, ethanol and ethyl acetate. Figure 2 shows the effect of different types of solvent on candlenut oil yield. The results show that the ethyl acetate produces highest yield of oil, 72.33% when compared with other solvents. Acetone produce the second highest yield with 46.65% followed by ethanol with 10.1% yield and methanol with 3.41%.

According to Cahyo et. al. the polarity index of ethyl acetate, ethanol, acetone and methanol was 4.3, 5.2 , 5.4 and 6.6, respectively [21]. It is interesting to note that polarity of the solvent used in the Soxhlet extraction had an influence on the yield, as the lower the polarity, the more the quantity of extract oil obtained. Ethyl acetate, a least polar solvent, provided the highest yield of 72.33 %, whereas the high polar solvent, methanol exhibited the lowest yield of 3.41%. This proves that the oil yield decreased as the relative polarity of the solvent increased. The oil from Candlenut seeds was classified as a non-polar lipid due to their major constituents of oleic acid[9]. Therefore, they dissolved better in ethyl acetate.

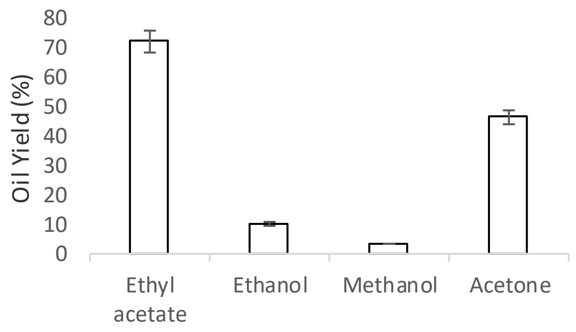

Fig 4.2. Effect of different type of solvent on candlenut (*Aleurites moluccana*) oil yield. (Conditions: extraction time3.5 hr, extraction temperature based on solvent boiling point, solid-to-liquid ratio 1:30 g/mL)

C. Chemical Constituents of Candlenut (Aleurites moluccana) Oil

The chemical constituents of the extracted candlenut oil were listed in Table1. From the GC-MS analysis, it can be seen that two major fatty acid content were found in the candlenut, which were palmitic acid that were found at retention time of 27.6679 min and oleic acid at 32.8558 min. Based on the previous studies conducted on the candlenut, it is proven that palmitic acid and oleic acid were abundantly present in the oil composition of candlenut[7]. Mogire (2013) also proves the existence of these fatty acids in the candlenut oil.

Fat is a basic macronutrient of the human diet and vegetable oils speak to all the more exceedingly consumed fat. The impacts of high-fat diet, mainly in saturated fatty acids (SFA), have been the concentration of a few dietary rules focusing on the reduction of cardiovascular disease (CVD), obesity-related diseases and, as of late, cancer prevention [22]. Palmitic acid ($C_{16:0}$) is the major saturated fatty acid in human milk, accounting for 17–25% of the total fatty acids [23].

Table 4.1. Chemical constituents of candlenut (Aleurites moluccana) Oil

It has been reported by two authors Ramadan et. al. and Ajayi & Adesanwo, 2009 that oils containing unsaturated fatty acids especially oleic acids can be used to lower plasma cholesterol. Oleic acid is very important in nervous cell construction as it has fundamental role in cardiovascular diseases prevention [26]. The high percentage of oleic acid in the oil makes it desirable in terms of nutrition and high stability cooking and frying oil [27]. Higher intake of oleic acid is associated with decreased risk of coronary heart disease caused by high cholesterol level in blood [28]. The ability of some unsaturated vegetable oils to reduce serum cholesterol level may focus attention on the oil to be very promising as edible oil [25]. People consuming the highest amounts of oleic acid were 89% less likely to have ulcerative colitis than those consuming the least amount of oleic acid [29].

No	**Retention time (min)**	**Compounds**
1	13.1804	Cyclotetradecane
2	15.6591	Benzaldehyde
3	15.7022	Neopentyl glycol
4	15.7597	1,3-Di(propen-1-yl) adamantane
5	16.0258	Pentanoic acid
6	27.4768	Dodecanoic acid
7	27.5200	Pentadecanoic acid
8	**27.6679**	**Palmitic acid**
9	27.6333	4-Heptadecyne
10	32.8270	Methoprene
11	**32.8558**	**Oleic aid**
12	32.8836	Methyltrans-4-(2-nonylcyclopentyl) butanoate
13	35.9316	Palmitic anhydride
14	37.8221	2,2-Difluoroheptacosanoic acid
15	39.1911	7-Methyl-Z-tetradecen-1-ol acetate
16	39.2276	Oleic anhydride

III. CONCLUSION

This study reported on the soxhlet extraction of oil from *Aleurites Moluccana*, which represent a promising alternative source of vegetable oil. The highest candlenut oil yield was obtained using ethyl acetate as a solvent with efficient extraction period of 3.5 hours for achieving optimum oil yield of 72.33%. The extracted soxhlet oil was found to contain oleic acid and palmitic acid.

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References

[1] M. R. Islam, M. Dalour, H. Beg, and S. S. Jamari, “Development of Vegetable-Oil-Based Polymers,” vol. 40787, 2014.

[2] E. O. Aluyor, C. E. Ozigagu, O. I. Oboh, and P. Aluyor, “Chromatographic analysis of vegetable oils : A review,” vol. 4, pp. 191–197, 2009.

[3] F. D. Gunstone, *Vegetable Oils in Food Technology*. 2011.

[4] D. Mogire, “Biodiesel production from candlenut and calodendrum capense seeds: process design and technological assessment,” 2013.

[5] P. Taylor and F. Karaosmanoglu, “Vegetable Oil Fuels : A Review Vegetable Oil Fu els : A Review,” no. December 2012, pp. 37–41, 2010.

[6] J. Fuls, C. S. Hawkins, and F. J. C. Hugo, “Tractor Engine Performance on Sunflower Oil Fuel,” no. April, pp. 29–35, 1984.

[7] A. Ahmad *et al.*, “Chemical Composition and Antioxidant Properties of Candlenut Oil Extracted by Chemical Composition and Antioxidant Properties of Candlenut Oil Extracted by Supercritical CO 2,” no. May 2011, 2016.

[8] Orwa et al, “Aleurites moluccana,” *Agrofor. Database 4.0*, vol. 0, no. 3, pp. 1–5, 2009.

[9] M. O. A. . Norulaini, Nik, Rahmad Setia Budi, Anuar Omar, MD Zaidul, “Major Chemical Constituents of Candlenut Oil Extract using Supercritical Carbon Dioxide,” *Pharm. Sci.*, vol. 2, no. 1, pp. 61–72, 2004.

[10] H. Ako, N. Kong, and A. Brown, “Fatty acid profiles of kukui nut oils over time and from different sources,” vol. 22, pp. 169–174, 2005.

[11] S. Salgın, D. Dinc, and G. Uluda, “The Journal of Supercritical Fluids Oil recovery in rosehip seeds from food plant waste products using supercritical CO 2 extraction,” vol. 118, pp. 194–202, 2016.

[12] W. Herchi, S. Bahashwan, K. Sebei, H. Ben Saleh, H. Kallel, and S. Boukhchina, “Effects of germination on chemical composition and antioxidant activity of flaxseed (Linum usitatissimum L) oil,” vol. 66, no. March, pp. 1–8, 2015.

[13] C. Figueiredo, J. Barroso, L. Pedro, and J. Scheefeer, “Factors affecting secondary metabolite production in plants: volatile components and essential oils,” *Flavour Fragr. J.*, vol. 22, no. November, pp. 206–213, 2007.

[14] A. M. D. Luque, “Soxhlet extraction of solid materials : an outdated technique with a promising innovative future,” vol. 369, 1998.

[15] Y. Li *et al.*, “Evaluation of alternative solvents for improvement of oil extraction from rapeseeds,” *Comptes Rendus Chim.*, vol.

17, no. 3, pp. 242–251, 2014.

[16] R. Dutta, U. Sarkar, and A. Mukherjee, "Extraction of oil from Crotalaria Juncea seeds in a modified Soxhlet apparatus : Physical and chemical characterization of a prospective," *Fuel*, vol. 116, pp. 794–802, 2014.

[17] P. A. Comet, "C-GC-MS , and its application to crude oil analysis," no. December, pp. 1–25, 1989.

[18] K. Andressa *et al.*, "The Journal of Supercritical Fluids Candeia (Eremanthus erythroppapus) oil extraction using supercritical CO 2 with ethanol and ethyl acetate cosolvents," vol. 128, no. January, pp. 323–330, 2017.

[19] K. Ramluckan, K. G. Moodley, and F. Bux, "An evaluation of the efficacy of using selected solvents for the extraction of lipids from algal biomass by the soxhlet extraction method," *Fuel*, vol. 116, pp. 103–108, 2014.

[20] W. Aida *et al.*, "Effect of ethanol concentration , extraction time and extraction temperature on the recovery of phenolic compounds and antioxidant capacity of Orthosiphon stamineus extracts," vol. 18, no. 4, pp. 1427–1435, 2011.

[21] A. Cahyo, M. Hasan, and H. Singh, "Effects of solvent properties on the Soxhlet extraction of diterpenoid lactones from Andrographis paniculata leaves," vol. 35, pp. 306–309, 2009.

[22] A. Mancini *et al.*, "Biological and Nutritional Properties of Palm Oil and Palmitic Acid: Effects on Health," pp. 17339–17361, 2015.

[23] F. Bar-yoseph, Y. Lifshitz, and T. Cohen, "Prostaglandins , Leukotrienes and Essential Fatty Acids Review of sn -2 palmitate oil implications for infant health," *Prostaglandins Leukot. Essent. Fat. Acids*, vol. 89, no. 4, pp. 139–143, 2013.

[24] B. M. F. Ramadan, M. M. S. Asker, and M. Tadros, "Lipid profile , antiradical power and antimicrobial properties of Syzygium aromaticum oil," vol. 64, no. 5, pp. 509–520, 2013.

[25] I. A. Ajayi and O. Adesanwo, "Comparative Study of the Mineral Element and Fatty Acid Composition of Dacryodes edulis Pulp and Seed," vol. 5, no. 3, pp. 279–283, 2009.

[26] I. A. Ajayi, D. S. Ajibade, and V. O. Taiwo, "RTICLES Short-term Toxicological Evaluation of," vol. 9, no. 1, pp. 67–72, 2013.

[27] B. F. Anwar, S. N. Zafar, and U. Rashid, "Characterization of Moringa oleifera seed oil from drought and irrigated regions of Punjab , Pakistan," vol. 57, no. 2, pp. 160–168, 2006.

[28] P. Corbett, "It ' s time for an oil change ! Opportunities for high-oleic vegetable oils," vol. 14, no. August, pp. 480–481, 2003.

[29] P. S. A. De Silva, R. Luben, S. S. Shrestha, K. T. Khaw, and A. R. Hart, "Dietary arachidonic and oleic acid intake in ulcerative colitis etiology : a prospective cohort study using 7-day food diaries," pp. 11–18, 1997.