Physicochemical properties of pili (*Canarium ovatum*) nut oil from Sabah, Malaysia

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ABSTRACT The information of pili (*Canarium ovatum*) nut oil from Sabah is still scare. Therefore, this study aimed to investigate the physicochemical properties of pili nut oil from Sabah. The oil was extracted using solvent extraction method which petroleum ether was the organic solvent. The extracted oil was evaluated for yield, color, slip melting point, iodine value, carotene content, fatty acid and triacylglycerol compositions. The oil was yellow in color with high yield (~70%) and semisolid state at room temperature (25 °C). The extracted oil had 18.5 °C for slip meting point and 96.4 g I_2/100 g for iodine value. The oil was rich in oleic, palmitic, stearic ad linoleic acids with the major triacyclglycerols were POO, PPO and OOO. The extracted oil was of good quality with free fatty acid content and peroxide value was about 1.5% and 2.5 mEq/kg oil, respectively. The β- carotene content of pili nut oil was about 66 ppm.

KEYWORDS: *Canarium ovatum*; Nut oil; Physicochemical properties; Fatty acid; Triacylglycerol
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INTRODUCTION

Pili (*Canarium ovatum*) belongs to the family Burseracea and indigenous to the Philippines. Pili trees also can be found in any other tropical regions including Malaysia and its bear fruits throughout the year. The Sabah Agriculture Department has planted pili trees for botanical research purposes. The pili fruit tree is one of the most under-exploited tropical Borneo trees. Therefore, not much research focused on the potential uses of pili fruits and nuts from Sabah. It is a deciduous tree measuring about 20-25 m in height and 40-50 cm in diameter. Pili fruits contain of pulp, shell and kernels or seeds known as nuts. In Philippines, pili is cultivated for its edible nut. Pili nuts can be eaten raw, roasted, or fried and are usually used in making confectioneries products such as cakes and ice cream (Pham & Dumandan, 2015; Coronel *et al*., 1996). The nut resembles a roasted pumpkin seed but when roasted it resembles pine nut with nutty flavour and waxy texture.

Cooking oil (also known as edible oil) is a plant or animal liquid fat used in frying, baking, and other types of cooking. Cooking oil is also used in food preparation and flavouring not involving heat, such as salad dressings and bread dips. Cooking oil is typically a liquid at room temperature, although some oils that contain saturated fat, such as coconut oil, palm oil and palm kernel oil are solid. Cooking oil such as palm oil which contains more saturated fats can withstand deep frying at higher temperatures and is resistant to oxidation compared to high polyunsaturated vegetable oils (Maszewska *et al*., 2018).

Solvent extraction will be used in this experiment as it results in higher yield compared to most other extraction methods although the time taken is longer compared to some extraction methods (López-Bascón *et al*., 2019). Applying heat to roast the pili nut is targeted to improve the extraction yield of the oil, reduce the moisture content, and inactivate lipase and other enzymes that promote the oxidation of the fatty acid. It also enhances cell tissue degradation to enhance easy flake penetration by the solvent used (Keneni *et al*., 2020). Solvent extraction can be carried out in several ways including the Soxhlet method. To date, no literature reported on extraction pili nut oil from Sabah using a Soxhlet method. Therefore, this study aimed to evaluate the physicochemical of pili nut oil from Sabah using a Soxhlet method.

METHODOLOGY

Sample Collection

Dried pili nut was obtained from Lagud Seberang Agriculture Research Centre, Sabah Agriculture Department, Tenom, Sabah, Malaysia. All chemicals used in this study were of general and analytical grades. All analyses were done in triplicate.

Oil Extraction

Oil extraction was done according to the method of AOAC (2005). Dried and ground pili nuts (150 g) were placed in a cellulose paper cone and the oil was extracted with petroleum ether (60 °C) in a 5 L Soxhlet extractor for 8 h. The oil was recovered using an Eyela N-1 rotary evaporator (Tokyo Rakakikal Co., Ltd, Tokyo, Japan). The extracted fat was placed in an oven at 60 °C for 1 h and then transferred into a capped bottle and stored at -20 °C until needed for analysis. Prior to analysis the oil was removed from frozen storage, left standing at room temperature for 1 h and then warmed at 60 °C until completely molten.

Determination of Oil Yield

According to AOAC (2005), the oil yield was calculated using Equation (1).

$$\text{Percentage of oil yield (w/w)} = W_{Oil}/W_{Sample} \times 100\% \quad (1)$$

W_{Oil} is the weight of extracted oil and W_{Sample} is the weight of the pili nut powder.

Determination of Colour

A colorimeter was used to determine the color of extracted oil based on the Hunter Lab system in terms of L* as the lightness of color from 0 (black) to 100 (white) in the tristimulus color coordinate system, a* ranges from negative (green) to positive (red). For b*, it ranges from negative (blue) to positive (yellow) (PORIM, 1995).

Determination of Slip Melting Point (SMP)

The SMP was conducted according to the PORIM test methods (PORIM, 1995).

Determination of Iodine Value (IV)

The IV was done according to the method of AOCS (AOCS, 2000).

Determination of Carotene Content

Carotene content was determined using PORIM test method (PORIM, 1995). About 0.1 g of oil sample were weighed and put into 25 mL volumetric flask. The sample was dissolved with a few milliliters of 2,2,4-trimethylpentane and diluted to marks. The solution was transfer to 1 mL of cuvette

and measure absorbance at 446 nm against solvent use. The blank sample was prepared according to the similar procedure. Analysis was conducted in triplicate. Carotene content is given in ppm of β-carotene and determined using Equation (2).

$$\text{Carotene} = 25 \times 3383/100w\ (a_s - a_b) \quad (2)$$

a_s is an absorbance of sample, a_b is a cuvette error and w is a weight of sample in grams (g).

Determination of Fatty Acid Composition

Fatty acid methyl esters (FAME) were prepared by dissolving 50 mg portion of oil in 0.8 mL of hexane and adding 0.2 mL portion of 1 M solution of sodium methoxide (AOCS, 2000), then analyzed on a gas chromatograph (GC) fitted with a flame ionization (FID) detector. The polar capillary column RTX-5 (0.32 mm internal diameter, 30 m length and 0.25 μm film thickness; Restex Corp., Bellefonte, 19 PA) were used. The oven temperature was programmed as follows: initial temperature of 50 °C (for 1 min), and programmed to increase to 200 °C at 8 °C/min. Both injector and detector temperatures were maintained at 200 °C throughout the analysis. The carrier gas (helium) flow rate was maintained at 1.0 /min with a split ratio of 58:1. The identification of the peaks of the samples were done with reference to a chromatographic profile containing FAME standards (Supelco, Bellefonte, PA). The percentage of FA were calculated as the ratio of the partial area to the total peak area (Yanty *et al*., 2018).

Determination of Triacylglycerol (TAG) Composition

The TAG composition was determined using liquid chromatography equipped with a refractive index as the detector. The analysis of TAG was performed on a RP-18 column (5 μm) (12.5 cm × 4 mm i.d.; Merck, Darmstadt, Germany). A mixture of acetone/acetonitrile (63.5:36.5, v/v) were used as a mobile phase and the flow rate were maintained at 1 mL/min. The oven temperature was maintained at 30 °C. The injector volume was set for 10 μL of 5% (w/w) oil in chloroform. Each sample were analyzed as triplicates, and the data were reported as area. The identification of the peaks of the samples were done using a set of TAG standards (Yanty *et al*., 2018).

Statistical Analysis

Statistical analysis was conducted using the Statistical Package for Social Sciences (SPSS) software (Version 28). All collected data from physicochemical analysis are assessed using independent t-test. Confidence level of 95% was used to determine if significant difference among mean score exist and results were done in triplicate.

RESULT AND DISCUSSION

The yield, colour, slip melting point, iodine value, carotene content are shown in Table 1. The yield of pili nut was 69.6%, which was higher as compared to other commercial vegetable oils such as groundnut (32 – 50%) (Ogunsola *et al*., 2021), sunflower seed (11.99 - 45.44%) (Moradi *et al*., 2018). The colour of pili nut oil was yellow in colour (L*= 13.27, a* = -1.63, b* = 16.52). The yellow colour in oil was related to carotene content that found in pili nut oil which was about 66 ppm. Carotenoids are antioxidants which important in human health by protecting cells and tissues from the damaging effects of free radicals and singlet oxygen (Zeb & Mehmood, 2004). According to Hathcock (2004), β-carotene is the most abundant form of provitamin A in fruits and vegetables and is currently incorporated in a wide variety of dietary supplements, including multivitamin, vitamin A and antioxidant formulations (Schierle *et al*., 2004). The carotene content in pili nut oil was higher as compared to corn oil (0.91 ppm) (Dauqan *et al*., 2011).

Table 1. Physicochemical properties of pili nut oil.

Parameter		Pili nut oil
Oil yield (%)		69.60 ± 0.94
Colour	L*	13.27 ± 0.16
	a*	-1.63 ± 0.12
	b*	16.52 ± 0.14
Carotene (ppm as β-carotene)		66.22 ± 0.75
Slip melting point (°C)		18.50 ± 0.50
Iodine value (g I_2/100 g)		96.4 ± 0.00

Each value in the table represents the mean ± standard deviation of three replicates.

Oil and fats do not have sharp melting points due to large amounts of different triglycerides (Marcus, 2013). The SMP of pili nut oil was 18.5 °C. The SMP of pili nut oil was found to be lower compared to those of other commercial oils and fats such as palm oil (30.5 °C) and cocoa butter (33-35.66 °C) (Alwi & Ming, 2019; Yanty *et al*., 2012). According to Tian *et al*. (2024), the number of carbon atoms in fatty acids eventually affects the SMP of the lipids. The SMP is affected by the fatty acid composition as a higher saturated fatty acid composition of oil will result in a higher melting point as more heat energy used to break the bond between fatty acids (Shin & Lee, 2022). The IV of pili nut oil was 96.4 g I_2/100 g. This value was higher as compared to palm oil (54 g I_2/100 g) and cocoa butter (34 g I_2/100 g) (Yanty *et al*., 2012). IV is related to degree of unsaturation of oil. Oils with high IV contain more unsaturated fatty acid. It is reported that oil that has high IV was more prone to oxidation (Norazlina *et al*., 2021).

Table 2 shows the fatty acid composition of pili nut oil. Pili nut oil contained oleic acid (41.64%) as a predominant fatty acid, followed by palmitic (30.38%), stearic (19.85%) and linoleic (7.01%) acids. The saturated and unsaturated ratio of pili nut oil was almost equal which made this oil became semisolid state at room temperature (25°C). Palm oil was also found to be in semisolid state at room temperature (25°C) due to 1:1 ratio of saturated and unsaturated fatty acid content (Mancini *et al*., 2015; Yanty *et al*., 2012). The saturated fatty acid (50.74%) was higher as compared to unsaturated fatty acid (49.26%). The major fatty acids of palm oil were palmitic (~44%), oleic (39%), linoleic (10%) and stearic (4.5%) acids (Mancini *et al*., 2015; Yanty *et al*., 2012). The variation in fatty acid composition could affect the SMP of oils because each fatty acids have different melting point.

Table 2. Fatty acid composition of pili nut oil.

Fatty acid	Percentage (%)
Palmitic acid (C16:0)	30.38 ± 0.07
Margaric acid (C17:0)	0.51 ± 0.00
Stearic acid (C18:0)	19.85 ± 1.66
Oleic acid (C18:1)	41.64 ±1.60
Linoleic acid (C18:2)	7.01 ± 0.01
Linolenic acid (C18:3)	0.61 ± 0.00
Saturated fatty acid	50.74
Unsaturated fatty acid	49.26

Each value in the table represents the mean ± standard deviation of three replicates.

The TAG composition of pili nut oil is shown in Table 3. The major TAG compositions of pili nut oil were POO (30.74%), PPO (19.49%) and OOO (12.17%). Nagai *et al*. (2020) and Yanty *et al*. (2012) reported that palm oil contained PPO and POO as the predominant TAG molecules with small amount of OOO. In palm oil, UStSt (47.97%) and UUSt (40.06%) are the most dominant TAG molecular groups (Yanty *et al*., 2012). On the hand, pili nut oil had UUSt (45.70%) as the highest

percentage followed by UStSt (37.30%). The TAG compositions are also dependent on major fatty acid as shown in Table 2.

Table 3. Triacylglycerol composition of pili nut oil.

Triacylglycerol	Percentage (%)
MMM	0.83 ± 0.22
MPL	2.54 ± 0.18
OOL	0.36 ± 0.02
MMP	2.86 ± 0.06
POL	6.73 ± 0.07
PPL	4.29 ± 0.02
OOO	12.17 ± 0.13
POO	30.74 ± 0.27
PPO	19.49 ± 0.10
PPP	0.49 ± 0.02
SOO	8.10 ± 0.02
PSO	9.30 ± 0.01
PPS	0.42 ± 0.06
SOS	1.68 ± 0.09
UUU	12.53
UUSt	45.57
UStSt	37.30
StStSt	4.60

Each value in the table represents the mean ± standard deviation of three replicates.
Abbreviations: M: Myristic; O: Oleic; P: Palmitic; S: Stearic; L: Linoleic; St: Saturated; U: Unsaturated.

CONCLUSION

Due to its relatively high oil content (~70%), pili nut oil (*Canarium ovatum*) represents a potential source of edible oil rich in oleic or monounsaturated fatty acid (41.64%). Oleic rich oils with high carotene content are susceptible to oxidation and would make this pili nut oil suitable as cooking oil. The semisolid state of the oil is resembled to palm oil, which could be further modified by fractionation process for producing many food products. For instance, the pili plantation is still limited in Malaysia as compared to other commercial crops such as oil palm. The implementation of extensive research regarding the properties of pili nut oil is required to pave the way for a prospective future plantation of pili and pili-based products particularly in Malaysia.

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