Physicochemical and quality characteristics of pili (*Canarium ovatum*) nut oil obtained from mechanical press extraction

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ABSTRACT Pili (*Canarium ovatum*) nut oil from Sabah, Malaysia was extracted using mechanical press extractor. The purpose of this study was to investigate the characteristics of pili nut oil in terms of physicochemical and quality properties. The yellowish oil had about 40% of oil content. The slip melting point and iodine value of the extracted pili nut oil were 19.5 °C and 96.75 g I_2/100 g, respectively. The major fatty acids found in the oil were oleic and palmitic acids. The major triacylglycerols were POO (28.01%) and PPO (20.58%). The extracted oil was found to be of good quality and had low peroxide content (2.56 meq/kg oil) and p-anisidine values (0.14 meq O_2/kg).

KEYWORDS: *Canarium ovatum*; Pili nut oil; Mechanical press; Physicochemical properties; Quality characteristics
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INTRODUCTION

Pili (*Canarium ovatum*) is a member of the Burseraceae family. The family Burseraceae, which includes over 100 species and is mostly found in Africa, Asia, and the Pacific Islands, consists of the huge genus *Canarium* (Azlan, 2009). Pili trees can be found in any other tropical region including Malaysia and they bear fruits throughout the year. Pili is one of the underused fruits in Malaysia, specifically in the East Malaysian states of Sarawak and Sabah. Therefore, not much research has focused on the potential uses of pili fruits and nuts from Sabah. The pili fruit is a drupe that is 2.3 cm to 3.8 cm in diameter, 4.0 cm to 7.0 cm in length and weighs between 15.7 g to 45.7 g. Pili fruits contain a pulp, shell and kernels or seeds known as nuts. Pili nuts are edible and can be used in many food products (Sangalang *et al*., 2023; De Jesus & Gamis, 2021; Aril-dela Cruz *et al*., 2018)

Compared to other oil extraction methods, mechanical press extraction is one of the non-hazardous, safe, green extraction methods specifically designed for the extraction of oil from oilseeds. Mechanical press does not involve the use of any heat, chemical and refining process, conserving the bioactive components of the oils. A high-quality oil may be produced by employing the mechanical press extraction at room or ambient temperature throughout the process. Additionally, these oil's natural and safe qualities appeal to consumers as they promote human health and help to avoid a variety of illnesses (Wandhekar *et al*., 2022). Due to its low production cost and ability to conserve a high concentration of bioactive components such as essential fatty acids, tocopherols, phenols, carotenoids and phytosterols, mechanical pressing is one of the most preferred methods of seed oil extraction by manufacturers and consumers (Parry *et al*., 2008). Therefore, the purpose of this study was to evaluate the physicochemical properties of extracted pili nut oil obtained from mechanical press extraction.

METHODOLOGY

Sample Collection

Mature pili fruits were obtained from the Sabah Agriculture Park, Tenom, Sabah, Malaysia. The pili fruits were washed, cleaned and dipped in hot water for about 5 - 10 min to ease the process of removing pili pulp from its shell following the method of Minh (2021) with slight modifications. The pili pulp was manually separated from the shell. After the pulp was removed from the shell, the shell was removed, washed and cleaned under running tap water to remove visible dirt. The shell was then crushed using a pestle of a mortar and the nut obtained was dried in an oven (TD-78T-SD, Thermoline, Australia) at 60 °C for 24 h (Hemphill & Martin, 1992). The dried nuts were ground using a Waring blender (Model HGBTWTS3, Dynamic Corporation of America, New Hartford, USA) and kept in a sealed bottle under cool dry storage. All chemicals used in this study were of general and analytical grades. All analyses were done in triplicate.

Oil Extraction

Pili nut powder was put in the cotton cloth and placed in the mechanical press extraction cup and manually pressed using a mechanical press machine to extract the pili nut oil using a pressure of 5 Pa at room temperature (25 °C). The extracted pili nut oil was put in a bottle, sealed and kept in the freezer at -20 °C until further analysis. The oil sample was taken out of frozen storage, thawed at room temperature for 1 h, and then heated to 60 °C until it was totally melted before analysis.

Determination of Oil Yield

According to Keneni *et al*. (2021), the oil yield was calculated using the following formula;

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

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

Determination of Slip Melting Point

The slip melting point was conducted according to the PORIM test methods (PORIM, 1995).

Determination of Iodine, Free Fatty Acid, Peroxide And p-Anisidine Values

The iodine value, free fatty acid, peroxide value and p-anisidine value values were determined according to the method of AOCS (AOCS, 2000).

Determination of Fatty Acid Composition

The fatty acid composition of oil was determined using a headspace autosampler (Model G1888, Agilent Technologies, Palo Alto, CA, USA) according to Fadzillah *et al*. (2019). The transfer line from the headspace sampler was directly connected to the injector of the gas chromatograph (GC). The oven was set at 110 °C. The extraction conditions in the headspace auto sampler were programmed as follows: 20 min for vial equilibration, 0.2 min for vial pressurisation, 0.2 min for filling the injection loop, 0.05 min for loop equilibration and 1 min for sample injection. Helium with a purity of 99.99% was used for vial pressurization and as the carrier gas. Volatile compounds were analysed using a GC-MS (Model 7890, Agilent Technologies, Palo Alto, CA, USA) equipped with a non-polar column

(J&W Scientific DB-5; 30 m, ID 0.25 mm, film thickness 0.25μm). The column temperature was kept at 40 °C for 10 min, increased at 6 °C/min to 240 °C and maintained isothermally for 20 min. The mass selective detector (Model MSD59556, Agilent Technologies, Palo Alto, CA, USA) was used in electron ionisation mode. A mass range between 30 and 550 m/z was scanned and the mass spectra obtained were compared to those of the National Institute of Standards and Technology (NIST) Mass Spectral Search Program for compound identification. Thirty-seven FAME standards (Sigma St. Louis, MO) were used as authentic samples to calculate the percentage of fatty acids based on peak area. The quantification of FAME was performed using a normalisation internal technique.

Determination of Triacylglycerol Composition

The triacylglycerol composition was determined using liquid chromatography equipped with a refractive index as the detector. The analysis of triacylglycerol 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) was 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 analysed in triplicates, and the data were reported as a percentage of area. The identification of the peaks of the sample was done using a set of triacylglycerol standards (Yanty *et al*., 2018).

Statistical Analysis

Statistical analysis was conducted using the Statistical Package for Social Sciences (SPSS) software (Version 28). All physicochemical analysis data were analysed using the independent samples t-test. A confidence level of 95% was used to determine if significant differences among mean scores exist. The mean ± standard deviation of three replicates were presented.

RESULTS AND DISCUSSION

In this study, pili nut oil obtained from mechanical press extraction was found to be yellow in colour with an oil yield of ~40% (Table 1). This value was lower as compared to the study of Zarinah *et al*. (2014) (~66%) although the same extraction method was used. This could be due to the differences between the mechanical press machine used and other factors such as pressure, pressing time, temperature and preheating time. Santoso *et al*. (2014) stated that pressure and pressing time significantly affect the oil yield while temperature and preheating time does not affect the oil yield significantly. Free fatty acid content, peroxide and p-anisidine values are general indicators of the quality of oil.

According to Table 1, the slip melting point and iodine value of pili nut oil were 19.50 °C and 96.75 g I_2/100g, respectively. The iodine value measures the unsaturation degree of oils. The iodine value of pili nut oil was lower as compared to those of commercial vegetable oils such as cottonseed (99-119 g I_2/100 g), corn (103-130 g I_2/100 g, soybean (120-143 g I_2/100 g), sunflower (110-143 g I_2/100 g), safflower (135-150 g I_2/100 g) and sesame (104-120 g I_2/100 g) oils, but higher than those of coconut (7.5-10.5 g I_2/100 g), olive (75-94 g I_2/100 g) and RBD palm (50-55 g I_2/100 g) oils (Malaysian Food Act, 1985). However, the iodine value of pili nut in this study were higher compared to the previous study by Zarinah *et al*. (2014). These values might be due to differences in fatty acid and triacylglycerol compositions of oils. Table 1 also shows the free fatty acid, peroxide value and p-anisidine value of pili nut oil which were 1.66%, 2.56 meq O_2/kg and 0.14 meq O_2/kg, respectively. Peroxide value of pili nut oil was higher than the acceptable limit (<0.3%) of edible oil according to the Codex (CODEX-STAN 210 - 1999) standard which might be due to the fact that the oil was still in the crude form. Zarinah et al. (2014) also found that the peroxide value of pili nut oil was higher than the acceptable limit by Codex. However, the peroxide and p-anisidine values of pili nut oil were within the acceptable limit (maximum 10 meq O_2/kg) of edible oil according to the Codex (CODEX-STAN 210 - 1999) standard. These results were also affected by initial composition, concentration of minor compounds with antioxidant properties, pro-oxidant characteristics, degree of processing, and storage conditions, which might cause different oxidative stability and deterioration of oils (Lisa *et al*., 2019).

Table 1. Physicochemical properties of pili nut oil obtained from mechanical press extraction.

Parameter	Pili nut oil[a]	Pili nut oil[b]
Oil yield (%)	39.73±0.62	65.69 ± 6.15
Colour	Yellow	-
Slip melting point (°C)	19.50 ± 1.32	-
Iodine value (g I_2/100g)	96.75 ± 3.15	32.44 ± 0.73
Free fatty acid (%)	1.66 ± 0.05	-
Peroxide value (meq O_2/kg)	2.56 ± 0.21	7.70 ± 0.02
p-Anisidine value (meq O_2/kg)	0.14 ± 0.03	-

[a]Each value in the table represents the mean ± standard deviation of three replicates.
[b]Zarinah *et al*. (2014).

Table 2 shows the fatty acid composition of pili nut oil. Pili nut oil contained oleic acid (45.69%) as a predominant fatty acid, followed by palmitic (32.77%), oleic (45.69%), linoleic (10.36%) stearic (10.35%), and linolenic (0.83%) acids. These values were comparable to the previous study by Zarinah *et al*. (2014). However, they found that pili nut oil contained palmitoleic acid (0.43%) which was not found in this study. They also stated that unsaturated fatty acid (62.04%) was higher than saturated fatty acid (37.96). Pili nut oil contained 56.88% and 43.12% of unsaturated and saturated fatty acids, respectively. The variation in fatty acid composition could affect the SMP and IV of oils because each fatty acid has a different melting point.

Table 2. Fatty acid composition of pili nut oil obtained from mechanical extraction.

Fatty acid	Pili nut oil[a] (%)	Pili nut oil[b] (%)
Palmitic acid (C16:0)	32.77 ± 3.96	28.19
Palmitoleic acid (C16:1)	-	0.43
Stearic acid (C18:0)	10.35 ± 0.85	9.34
Oleic acid (C18:1)	45.69 ± 2.03	50.70
Linoleic acid (C18:2)	10.36 ± 1.65	10.61
Linolenic acid (C18:3)	0.83 ± 0.33	0.73
Saturated fatty acid	43.12	37.96
Unsaturated fatty acid	56.88	62.04

[a]Each value in the table represents the mean ± standard deviation of three replicates.
[b]Zarinah *et al*. (2014).

The triacylglycerol composition of pili nut oil is shown in Table 3. The major triacylglycerol compositions of pili nut oil were POO (28.01%) and PPO (20.58%). According to Yanty *et al*. (2012), palm oil also contained PPO (31.61%) and POO (24.76%) as the major triacylglycerol molecules. The triacylglycerol composition is also dependent on the major fatty acids (oleic and palmitic acids) as shown in Table 2. The triacylglycerol composition could also affect the slip melting point and iodine value of oils because each triacylglycerol molecule has its own melting point (Nusantoro *et al*., 2016).

Table 3. Triacylglycerol composition of pili nut oil.

Triacylglycerol	Percentage (%)
LnLnL	0.86 ± 0.22
LnLL	0.42 ± 0.13
LLL	0.59 ± 0.00
PLnL	0.46 ± 0.17
OLL	0.97 ± 0.03
OOL	2.36 ± 0.04
POL	6.79 ± 0.12
PPL	4.70 ± 0.02
OOO	9.43 ± 0.03
POO	28.01 ± 0.30
PPO	20.58 ± 0.16
PPP	0.53 ± 0.11
OOS	8.09 ± 0.19
POS	1.68 ± 0.09
PPS	0.60 ± 0.03
SSO	2.12 ± 0.03
Unknown	0.64 ± 0.02

[a]Each value in the table represents the mean ± standard deviation of three replicates.
Abbreviations: O: Oleic; P: Palmitic; S: Stearic; L: Linoleic; Ln: Linolenic.

CONCLUSION

Pili nut oil (*Canarium ovatum*) represents a potential source of edible oil rich in oleic and palmitic acids which can be used as specialty or cooking oils. The ratio of unsaturated to saturated fatty acids in pili nut oil almost resembles that of palm oil. The quality of oil in terms of free fatty acid content could be reduced through refining process.

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