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Preparation and Characterisation of Indigenous Tengkawang (Shorea Stenoptera) Butter for Food and Cosmetics Material

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Abstract: Tengkawang is an indigenous plant that has potential as a source of vegetable fat. The quality of tengkawang fat is traditionally low. This study aims to reduce free fatty acid (FFA) and physicochemical analysis of tengkawang fat and compare it with shea butter. The purification process of tengkawang includes degumming, neutralization, and bleaching. The FFA levels (%) before and after purification in tengkawang and shea butter were 7.75 and 2.28 for tengkawang butter and 0.47 and 0.18 for shea butter. Peroxide numbers (meq O_2 / kg sample) before and after refining in tengkawang butter and shea butter were 9.54 to 3.61 for tengkawang and 10.31 to 4.84 for shea butter. The saponification number of tengkawang butter was higher than shea butter, while the iodine number of tengkawang butter was lower than shea butter. The fatty acids in tengkawang butter are dominated by oleic acid, stearic acid, and palmitic acid, while the fatty acids in shea butter are dominated by oleic acid, stearic acid, and a little linoleic acid. After the refining process, tengkawang fat's quality complies with the Indonesian Raw Material Trade Standard (SNI 2903: 2016). This study indicates that tengkawang butter can be used as a raw material for the food and cosmetic industry.

Keywords: tengkawang butter, free fatty acid, shea butter.

用于食品和化妆品材料的本土登卡旺(短翅目)黄油的制备和表征

摘要:腾卡旺是一种本土植物,具有作为植物脂肪来源的潜力。腾卡旺脂肪的质量传统上很低。本研究旨在降低腾卡旺脂肪的游离脂肪酸(FFA)和理化分析,并将其与乳木果油进行比较。腾卡旺的提纯过程包括脱胶、中和、漂白。在腾卡旺和乳木果油中纯化前后的FFA水平(%)分别为7.75和2.28,腾卡旺黄油为0.47和0.18。腾卡旺黄油和乳木果油精炼前后的过氧化物值(梅克O2/公斤样品)分别为9.54至3.61和10.31至4.84的乳木果油。腾卡旺黄油的皂化值高于乳木果油,而腾卡王黄油的碘值低于乳木果油。腾卡旺黄油中的脂肪酸以油酸、硬脂酸和棕榈酸为主,而乳木果油中的脂肪酸以油酸、硬脂酸和少量亚油酸为主。提炼后的腾卡旺油脂质量符合印尼原料贸易标准(SNI2903:2016)。该研究表明,腾卡旺黄油可作为食品和化妆品行业的原料。

关键词:腾卡旺黄油、游离脂肪酸、乳木果油。

1. Introduction

Despite being an agricultural country, Indonesia still imports vegetable fats as raw materials for its cosmetics and food industry. Based on the Central Statistics Agency (BPS) data in 2018, Indonesia imported 20,880,060 kg of solid vegetable fat to meet

domestic needs [1]. One vegetable fat source from African countries such as Ghana, Nigeria, and Kenya is Shea butter for cosmetics raw material [2]. Shea butter is widely used as a raw material for cosmetics because of its high fatty acid content [3]. To reduce the dependency on overseas sourcing, local vegetable fats

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that can replace part or all of the imported raw materials are needed. One of the candidates is tengkawang fat (Shorea stenoptera), an endemic plant for Kalimantan. Tengkawang fruit is produced from several types of plants belonging to the Dipterocarpaceae family [4].

In the world of trade, tengkawang fruit is known as the illipe nut or Borneo tallow nut. During the harvest season, productive tengkawang trees with an age range of 30 to 100 years can produce fruit as much as 250-400 kg per tengkawang tree [5]. The fat content of tengkawang seeds varies depending on the type and quality of the seeds. Generally ranging from 43 to 61%, while according to Ketaren, the fat content of tengkawang reaches 50 to 70 % [6]. The properties of tengkawang fat are similar to cocoa butter, classified as cocoa butter substitutes, and can be used in the cosmetic industry [7]. Another advantage is that the price of tengkawang fat is lower than cocoa butter [8]. The community widely uses Tengkawang oil as a raw material for cooking oil, margarine, or further processed into a chocolate processing mixture [6]. In 2008, the people of Embaloh Hilir Subdistrict, Kapuas Hulu District, received a profit of 1,405,685,000/harvest or an average value of IDR 10,812,962/household/harvest [9].

In 2015, local communities in the Bengkayang area, West Kalimantan, produced 700 kg of processed dried tengkawang seeds [10]. The processing of tengkawang fat is traditionally carried out using a device known as an "apit" with a production capacity of 4 - 5 kg of fat in one operation. Research conducted by Hidayat et al. purified tengkawang fat using activated carbon [11]. Muhammad et al. performed the purification of tengkawang fat with thermally activated bentonite and succeeded in reducing beta carotene levels, acid numbers, and peroxide numbers [12]. The purification process with thermal and acid bentonite can improve the quality of tengkawang fat [13]. Ramadhani et al. used lignin to reduce free fatty acids and beta carotene content in tengkawang butter [14]. However, there is a need to characterize the physical properties of tengkawang butter further to comply with Indonesian standards (SNI) 2903: 2016.

This research aims to identify and improved the physicochemical Peroxide, content (FFA, saponification, and iodine numbers) of the indigenous tengkawang butter and compare it with shea butter as a raw material for the cosmetic and food industry. Therefore, it is essential to characterize and improve the quality of tengkawang butter to achieve Indonesian standards (SNI) as industrial material standards. The traditional practice has a unique way of processing tengkawang butter. The quality of tengkawang butter is improved by reducing FFA and peroxide numbers to follow the Indonesian standard (SNI) 2903: 2016. This research will increase the potential of tengkawang fat as a raw material for the Cosmetic and food industry.

2. Materials and Methods

2.1. Materials

Tengkawang fat was obtained from Nanga Yen Village, Kapuas Hulu Regency, West Kalimantan, Indonesia. The shea butter was imported from Ghana. Chemicals such as NaOH, H₃PO₄, KI, Na₂S₂O₃, KIO₃, HCl starch were obtained from Merck Millipore (Germany). Bentonite was obtained from Lampung Regency, Lampung Province. The tengkawang and shea butter were purified by degumming, neutralization, and bleaching.

2.2. Purification of Tengkawang and Shea Butter

The purification process of tengkawang and shea butter was done by degumming with H_3PO_4 , neutralizing NaOH, and bleaching with bentonite. The refining process was carried out based on the method performed by Hidayat et al. [11] and Darmawan et al. [13].

The degumming process used 20% H3PO4 as much as 1% (w/w). The mixture was then stirred using a magnetic stirrer for 30 minutes in a beaker glass. The fat is then separated from the impurities using warm water at 60-70°C.

The neutralization process was carried out using 10% (w/w) 1 M NaOH and added to the degummed butter. The mixture of fat and base was then stirred with a magnetic stirrer for 30 minutes at a temperature of 60-70°C. The soap layer formed was separated by centrifugation. Warm water was used to remove any soap residue from the mixture.

The bleaching process was performed with 5% bentonite (w/w) into degummed and neutralized butter. The mixture of bentonite and fat is then stirred for 30 minutes at a temperature of 60-70°C. After the stirring process, the bentonite was separated from the fat using the vacuum filtration method.

2.3. Chemical Analysis of Tengkawang and Shea Butter

Thermal properties of the fat samples were determined using Differential Scanning Calorimetry (DSC) instrument. A total of 1 gram of sample is put into the DSC container. The sample was heated to 70°C with a heating rate of 1°C/min and allowed to stand for 30 minutes. Afterward, the sample is cooled to a temperature of 10°C. The data generated from the thermogram will be compared to determine changes in the thermal properties.

Fatty acid content was determined using a gas chromatography-mass spectroscopy (GC-MS) instrument. As much as 1 gram of each fat sample, i.e., tengkawang fat and shea butter, was prepared and dissolved in 100 mL heptane solvent. 1μ L of each fat solution was injected into the GC column. Before the fat sample was injected, the mobile phase must be

ensured to run well. The mobile phase used in this study was helium gas. After heating to a temperature of 300°C, the MS detector will detect the vaporized component to produce a chromatogram of separation, and the mass ratio of the sample components could be determined.

Fourier transform infrared spectroscopy (FTIR) instrument was used to assess the functional groups in the fat. The test was carried out following the SOP of the Perkin Elmer 100 Perkin Elmer Spectrophotometer tool Model: Frontier S/N: 96772 in the wavenumber range 4000 cm⁻¹ to 600 cm⁻¹, with ten readings and a resolution of 8 cm⁻¹. The resulting spectrum was analyzed using Perkin Elmer's Spectrum Software FT-IR software.

2.4. Quality Analysis of Tengkawang and Shea Butter

The quality of tengkawang and shea butter was analyzed based on their free fatty acids, peroxide number, iodine number, and melting point. Determination of FFA, peroxide, saponification, and iodine numbers were based on Indonesian standards (SNI) [15].

Analysis of free fatty acids was carried out by alkaline titration using 0.1 M NaOH as the titrant. The calculation of the FFA was based on the following equation:

Free fatty acids (%) =
$$\frac{VxNxM}{10m}$$
 (1)

where:

V - volume of NaOH (mL);

N - normality of NaOH (N);

m - mass of butter (g);

M - relative mass of fatty acid as oleic acid (g/mol).

Peroxide number was analyzed by iodometry using $Na_2S_2O_3$ as the titrant and starch as the indicator. The peroxide number was calculated based on equation 2.

$$Peroxide\ number = \frac{(Vb - Vs)xN}{m} \tag{2}$$

where:

Vb - Titrationvolume of $Na_2S_2O_3$ 0.1 N for blank titration (mL);

V_s - Titrationvolume of Na₂S₂O₃ 0.1 N for sample (mL);

N - Normality of Na₂S₂O₃ solution (0.1 N);

m - the mass of the sample (gram).

Iodine number was assessed using the iodometric titration method to determine the double bond in the sample. The reagents used were Wijs solution as the reagent, sodium thiosulfate solution as the titrant, and starch as an indicator. The equation for the iodine number is:

$$Iodine\ number = \frac{12,69x\ N\ x\ (V_0 - V_1)}{m}$$
(3)

where:

N - Normality of Na₂S₂O₃ 0.1 N;

 V_0 - Titrationvolume of $Na_2S_2O_3$ solution 0.1 N for blank (mL);

 V_1 - Titrationvolume of $Na_2S_2O_3$ 0.1 N for sample (mL); m - mass of butter (g).

Saponification numbers were analyzed using acidbase titration to determine which fat components can undergo saponification reactions. The equation for saponification number is:

Saponification number =
$$\frac{56.1 \times N \times (V_0 - V_1)}{m}$$
 (4)

where:

N - normality of HCl 0.5 N;

Vo - titration volume of HCl 0.5 N for blank (mL);

V1 - titration volume of HCl 0.5 N for sample (mL);

m - mass of butter (g);

56.1 - the molecular weight of KOH, in g⋅mol⁻¹.

Lastly, the slip melting point (SMP) test was performed to determine the sample's melting point. The sample was inserted into a capillary tube where one end was closed with a flame. The capillary tube containing the sample was immersed into the paraffin liquid in the melting point apparatus equipped with a magnetic stirrer. The temperature recorded is the temperature at which the sample begins to melt.

3. Results and Discussion

3.1. Fatty Acids Analysis of Tengkawang and Shea Butter

The fatty acid analysis was performed to get the fatty acid composition of tengkawang and shea butter. Fig. 1 is a GC chromatogram of tengkawang butter.

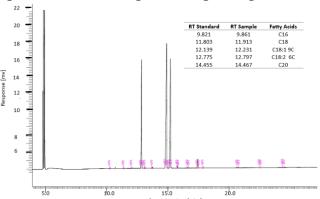


Fig. 1 GC chromatogram of tengkawang butter

Based on the GC chromatogram, the peaks that appeared on tengkawang butter were palmitic acid (C16), stearic acid (C18), oleic acid (C18: 1), linoleic acid (C18: 2), and arachidic acid (C20) with RT values of 9.861, 11.913, 12.231, 12.797, 14.467 respectively. The peak is dominated by stearic acid, oleic acid, and palmitic acid in tengkawang butter. The sample's RT value can be compared with the standard RT value to obtain the fatty acid composition [16].

RT standard of palmitic acid (C16), stearic acid (C18), oleic acid (C18: 1), linoleic acid (C18: 2), and arachidic acid (C20) 9.821, 11.803, 12.139, 12.775, and 14.455, respectively.

A fatty acid analysis was carried out to determine fatty acid composition between tengkawang and shea butter. Fig. 2 is a GC chromatogram of shea butter.

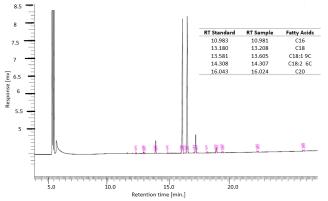


Fig. 2 GC chromatogram of shea butter

Based on the GC chromatogram, the peaks that appear in the shea butter are palmitic acid (C16), stearic acid (C18), oleic acid (C18: 1), linoleic acid (C18: 2), and arachidic acid (C20) with RT values of – 10.981, 13.208, 13.605, 14.307 and 16.024,

respectively. The dominant peaks that appear are the peaks of stearic acid and oleic acid. There is a significant difference between the peaks on tengkawang butter and shea butter, namely the absence of high peaks in shea butter. Also, there are differences in the RT value of the samples in tengkawang butter and shea butter. This phenomenon is probably due to differences in the composition of tengkawang butter and shea butter.

For determining the fatty acids composition, a comparison between the RT sample and the standard RT was carried out to obtain the respective composition of the fatty acids in tengkawang butter and shea butter. The fatty acid composition obtained was then compared with various fat sources to determine the potential of tengkawang butter and shea butter. Table 1 shows the fatty acid data for various sources of fat.

Table 1 Fatty acid composition from various butter sources

Fat Resource	Fatty Acid Composition						Ref.		
	C12:0	C14:0	C16:0	C18:0	C18:1	C18:2	C18:3	C20:0	•
S. pinanga	-	0.03	11.78	1.560	42.79	22.04	-	-	[17]
S. mecisopteryx	-	0.04	14.51	0.80	31.28	27.05	-	-	[17]
Cocoa butter	-	-	25.60	36.60	32.70	2.80	-	-	[18]
CBE	-	-	27.00	33.00	35.00	3.00	-	2.00	[19]
Sintang TB	0.15	0.08	20.22	42.60	31.29	1.01	0.45	3.53	[13]
Shea butter	-	-	3.14	43.12	44.82	5.98	0.09	1.70	This study
Tengkawang Butter	-	0.05	17.74	43.67	32.68	0.89	0.12	1.91	This study

Based on the data in Table 1, there is a difference in composition between shea butter and tengkawang butter. In tengkawang butter, the fatty acid components are dominated by stearic acid, oleic acid, and palmitic acid, while in shea butter, it is dominated by oleic and stearic acids. The compositions of stearic acid, oleic acid, and palmitic acid were 17.74, 43.67, and 32.68 for tengkawang and 3.14, 43.12, and 44.82 shea butter. This difference is due to the different types of tengkawang butter and shea butter. In this study, the tengkawang sample used was Shoreastenoptera, and the shea butter sample was Vitellaria paradoxa. The composition's difference can be seen in the differences in chemotaxonomy between tengkawang butter and shea butter. Chemotaxonomy or chemical taxonomy is used to classify plants based on their chemical components [20]. Plants that have different types of genera will have different chemical compositions.

The tengkawang used in this study came from Nanga Yen village. Different growth sites can produce slightly different fatty acid compositions. Also, the process carried out is different in the regions of origin. Based on previous research, tengkawang fat processing in the Nanga Yen area uses a solar heating process [13]. Apart from *S. stenoptera*, tengkawang has other types, namely *S. pinanga* and *S. mecisopteryx*. The fatty acid composition of *S. stenoptera*, *S. pinanga*, and *S. mecisopteryx* was also different due to different types of tengkawang. Gusti and Zulnely conducted

research using *S. pinanga*, and *S. mecisopteryx* has a dominant fat composition in oleic acid, linoleic acid, and palmitic acid [17].

The fatty acid composition in tengkawang is close to cocoa butter and cocoa butter equivalent (CBE). Gunstone stated that tengkawang fat could be used to make cocoa butter equivalent [21]. Traditional communities in Kalimantan use fat as a food source to meet their fat needs, such as making black butter rice [22]. Because it is high in saturated and unsaturated fatty acids such as stearic acid, palmitic acid, and oleic acid, tengkawang is also used to make lipsticks [23] and wet noodles [24].

3.2. FTIR Analysis of Tengkawang and Shea Butter

FTIR analysis was performed to determine the functional groups contained in tengkawang and shea butter. Fig. 3 shows the spectra of tengkawang butter and shea butter.

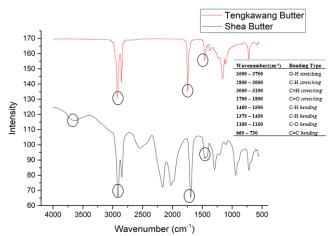


Fig. 3 FTIR spectra of tengkawang and shea butter

Based on the spectra in Fig. 3, the high peaks at the wavenumbers around 2900 - 3000 cm⁻¹ and 2852 cm⁻¹ show the stretching of the C-H bond on the methylene (CH₂) group. Then there is a tiny peak at the wave number $3000 - 3100 \text{ cm}^{-1}$, which shows the C = H bond's stretching in the fatty acid double bond. The high and sharp peak at wave number 1744 cm⁻¹ is the carbonyl bond stretching (C = O). The carbonyl bond is a characteristic feature of the spectrum of fatty acid compounds or fatty acid esters. A wavenumber 1159 cm⁻¹ shows the stretching of the C-O bonds in the ester contained in the triglycerides. It can be seen that there is a distinctive peak at the wavenumber around 3750 -3500 cm⁻¹, which is a characteristic of the O-H functional group in the shea butter spectra group. The difference between tengkawang fat and shea butter is the presence of O-H peaks in the shea butter. There may be compounds containing the (O-H) group in the shea butter so that there is an O-H peak that is wide enough at the wavenumber above 3500 cm⁻¹.

The appearance of O-H peaks in shea butter is probably due to the phenolic compound content. Maranz et al. (2003) proved that shea butter has phenolic content such as gallic acid, catechin, epicatechin, epicatechin gallate, and quercetin [25]. The absence of O-H peaks in tengkawang butter is probably due to the loss of phenolic compounds in the traditional tengkawang fat extraction process.

3.3. Thermal Analysis of Tengkawang and Shea Butter

TGA-DSC characterization was carried out to determine the thermal profile of tengkawang butter and shea butter. Fig. 4 is the TGA thermogram of tengkawang butter and shea butter.

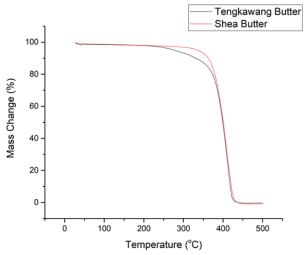


Fig. 4 TGA thermogram of tengkawang butter and shea butter

Based on Fig. 4, there are differences in the thermogram of tengkawang butter and shea butter. In tengkawang butter, if the temperature has reached 300 °C, the mass change is close to 90%, while in shea butter, it is still above 95%. This is because shea butter has more C18 fatty acids (stearic acid and oleic acid) than tengkawang butter. So that tremendous heat energy is needed for the decomposition of these fatty acids into smaller carbon compounds. The fatty acid decomposition reaction involves radical reactions such as peroxide radicals, hydroxide radicals, ozone, nitrates, and DPPH [26-29]. Radical compounds will attack the double bonds in unsaturated fatty acids to form new radical compounds or break the double bonds if the energy given is large enough to break the double bond. Fatty acids whose double bonds are broken will be decomposed into short-chain compounds, which are more volatile than long-chain fatty acids. As a result, there will be a large mass change on the thermogram detector so that the mass change will be smaller.

The DSC thermogram shows the endothermic melting event of tengkawang and shea butter. Fig. 5 is a DSC thermogram of tengkawang and shea butter.

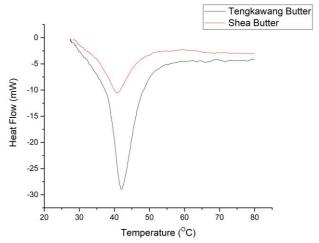


Fig. 5 DSC thermogram of tengkawang and shea butter

Based on Fig. 5, the temperature of shea butter is lower than tengkawang butter. This phenomenon is due

to the composition of shea butter dominated by unsaturated fatty acids such as oleic acid. Unsaturated fatty acids have a lower melting point than saturated fatty acids on the same carbon chain. Oleic acid (C18: 1) has one double bond, so it has a melting point at a temperature of 10-16°C [21, 30]. Stearic acid (C18: 0) is a saturated fatty acid with a melting point of 68-71°C [21, 30]. Because the composition of shea butter is mostly oleic acid and stearic acid, shea butter's melting point is lower than tengkawang. Table 2 is the enthalpy data from various fat sources.

Table 2 Enthalpy data from various sources

Fat Resources	Temperature (°C)		Enthalpy (J/g)	Reference	
	Onset	Offset		_	
Cocoa Butter	25.97	37.70	116.20	[31]	
Rambutan fat	34.87	38.95	113.70	[32]	

Dark chocolate	12.54	32.32	121.52	[33]
CBE	26.20	34.60	30.70	[33]
Shea butter	7.13	17.82	58.93	[34]
Shea butter	34.90	47.60	56.28	This study
Tengkawang	34.7	48.40	91.07	This study
Butter	34.7	46.40	91.07	

Based on Table 2, the enthalpy value of tengkawang fat is closer to dark chocolate fat and brown fat. This phenomenon is probably because the fatty acid composition is almost the same between tengkawang fat and brown fat. The enthalpy in shea butter is smaller than tengkawang because saturated fatty acids dominate the fatty acid component of shea butter. This enthalpy data supports the component data of the fatty acid profile in shea butter.

Table 3 Quality parameter of tengkawang and shea butter

Parameter	Unit	Tengkawang standard [35]	Shea standard [36]	TBI	SBI	TBR	SBR
Free fatty acid	%	Max 3.5	1	7.75	0.47	2.28	0.18
Saponification Number	mg KOH/fat	189 - 200	160-195	188	180	188	181
Iodin Number	g I ₂ /100 g	25 - 38	30 - 75	31.5	61.26	31.6	61.62
Melting Point	°C	35 - 39	35 - 40	28	33	27	34
Peroxide Number	meq O ₂ /100 g	Max 10	Max 10	9.54	10.31	3.61	4.84

Note: TBI - tengkawang butter initial, SBI - shea butter initial, TBR - tengkawang butter refined, SBR - shea butter refined

Based on Table 3, there are differences in the values of free fatty acid, peroxide number, melting point, and iodine number. This difference may be due to differences in the processing of tengkawang fat and shea butter. The tengkawang fat process is still carried out using a traditional process to make the fat's quality relatively low. Besides, there are differences in the composition of the constituent fatty acids, affecting the value of the quality parameters of tengkawang butter and shea butter.

The free fatty acid of shea butter is very low compared to tengkawang fat, namely 0.47 for shea butter and 7.75 for tengkawang butter. This is due to the different types of fat, the place of growth, and the tengkawang and shea butter production process. After the purification process, tengkawang free fatty acid decreased significantly to 2.28, while in shea butter, it decreased to 0.18. This is due to the neutralization process, which reduces free fatty acids to form soap. Soap as a by-product can also be used as a cosmetic.

The two fats' peroxide numbers are still high because it is still around the maximum value of 10.31 and 9.43 for shea butter and tengkawang butter. The high number of peroxide is due to the many impurities that are in the fat component. The purification process reduced the peroxide numbers in tengkawang butter to 3.61 and 4.84 for shea butter. The decrease in peroxide number occurs after the bleaching process because the impurities undergo an adsorption process on the bentonite. Previous studies have shown that the

bleaching process using bentonite can reduce peroxide numbers [13] and beta carotene [12].

The iodine numbers of tengkawang butter differ significantly when compared to shea butter. The iodine numbers for shea butter and tengkawang butter are 61.26 and 31.5, respectively. This is because the unsaturated fatty acid content in shea butter is more significant than tengkawang fat. This can be seen in the fatty acid composition of shea butter and tengkawang butter. In shea butter, oleic acid and linoleate components are higher than tengkawang butter, so the iodine of shea butter is higher than tengkawang butter. High levels of oleic and linoleic acids make fats susceptible to oxidation degradation due to high light and temperature [17]. This shows that tengkawang fat has higher oxidative stability than shea butter.

The saponification numbers show how many mg of KOH is needed to lather 1 g of fat. The saponification number calculates the content of the ester bonds in the fat [37]. The saponification numbers also calculate the chain length of the fatty acids in the ester bonds. The longer the fatty acid chain, the smaller the saponification number. The fatty acid component of tengkawang butter is dominated by palmitic acid, oleic acid, and stearic acid, while shea butter is dominated by oleic acid and stearic acid. This makes the saponification numbers of tengkawang fat higher than shea butter.

4. Conclusion

This study succeeded in reducing free fatty acids and physicochemical characterization of tengkawang butter. Free fatty acid (%) in tengkawang butter decreased from 7.75 to 2.28, while in shea butter, it decreased from 0.47 to 0.18. The peroxide number (meq O2/kg sample) in tengkawang butter decreased from 9.54 to 3.61, while in shea butter, it decreased from 10.41 to 4.84. Tengkawang butter iodine number is lower than shea butter. The saponification numbers of tengkawang butter are higher than shea butter. The quality of tengkawang butter is complying with the Indonesian Trade Standard for tengkawang (SNI 2903:2016). The main component of fatty acids in tengkawang butter are oleic acid, palmitic acid, and stearic acid, while shea butter is oleic acid, stearic acid, and minor linoleic acid. This study shows that the quality of tengkawang after purification can meet the Indonesian Trade Standard for tengkawang and can be a raw material for food and cosmetics.

Acknowledgments

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