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Tamanu and coconut oil blends for soap making from extraction of Tamanu kernel with coconut milk

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Abstract

This is a study on the extraction of oil from raw tamanu (*Calophyllum inophyllum L.*) kernel with milk from fresh coconut (*Cocos nucifera L.*) to obtain a blend (TCO) of these. The weight ratios of raw tamanu kernel (RTK) and grated coconut meat (GCM) were varied and fatty acid compositions were assessed. The RTK/GCM ratios were 0.15, 0.25 or 0.35 in 1.0 kg batches of RTK and GCM heated to 70 °C in a water bath. The yield of mixed oil was 14.7, 16.1 and 18.0 for the RTK/GCM ratios of 0.15, 0.25 and 0.35, whereas the yields of pure tamanu oil (TO) and pure coconut oil (CO) were 31.6 and 18.3, respectively. The ratios of unsaturated fatty acids (UFA) to saturated fatty acids (SFA) were 30:70, 40:60 and 51:49 for the respective RTK/GCM ratios 0.15, 0.25 and 0.35. Saponification number, iodine value, and acid value were determined for the extracts. The results indicate the RTK to GCM ratio 0.25 as advantageous, giving an appropriate UFA:SFA ratio of 40:60. Soaps made from CO were assessed and a target TCO was investigated. The soap properties tested satisfied the Thai Community Product Standard of soap (TCPS 94-2546) and the Thai Industrial Standard TIS 29-2545. The TCO soap had better properties than the CO soap as regards pH, hardness, moisture content, and total fatty matter.

Keywords: Tamanu oil, Coconut oil, Co-extraction, Soap, Fatty acid

1. Introduction

Cold pressed extraction is the preferred process for obtaining coconut oil by squeezing the oil out of dried coconut meat or copra. It is suitable for industrial scale operations, while hot extraction of fresh coconut milk is suitable for cottage level operations. The product is used in cooking and in body care. Coconut oil (CO) soap is also popular among consumers because coconut oil contains mainly lauric acid, which has outstanding properties in reducing inflammation and skin infections, and kills bacteria on the skin [1]. Coconut soap is a well-known community product in Thailand, but pure coconut oil soaps have inferior properties: the soap texture is quite hard and this causes the skin to become dry and irritated. Therefore other types of oil should be mixed in, especially such that are high in unsaturated fatty acids. The disadvantages of CO based soap stem from CO containing more than 90% saturated fatty acids (SFA). According to Benjamin and Abbass [2] the proportion of SFA in the oil for soap production should be around 60 percent. Reducing the amount of SFA decreases alkaline usage in soap production and the soap will moisturize skin. This also reduces hardness of the soap texture and the risk of skin irritation. Therefore, the oil mix for soap should have a component with high content of unsaturated fatty acids (UFA) to compensate for the high SFA in coconut oil. The kernel from tamanu fruit contains more than 68% of UFA. The unsaturated oil of tamanu is mostly oleic and linoleic, which can moisturize and maintain skin integrity according to Lin et al. [3], and will beneficially alter soap properties from those achieved with coconut oil alone. This is a study on extracting oil from tamanu seeds with coconut meat to obtain a suitable ratio of SFA to UFA for making high-quality soap. The extraction used fresh raw materials from coconut and tamanu fruits without any chemical solvents. The raw materials were obtained from households in a rural beach area in Thailand, in which coconut and tamanu trees are abundant as parts of native flora.

2. Materials and methods

2.1 Raw material

The raw materials used in this work were ripe fruits of tamanu and coconut. The fruits were collected from Hua Sai district in Nakhon Si Thammarat province located on the Pacific shore of Southern Thailand, as shown in Figure 1. The kernels from tamanu seeds were mechanically crushed to 1-2 mm size, whereas the coconut meat was grated. The grated coconut meat was squeezed after mixing with warm distilled water, filtering off the grated coconut solids to

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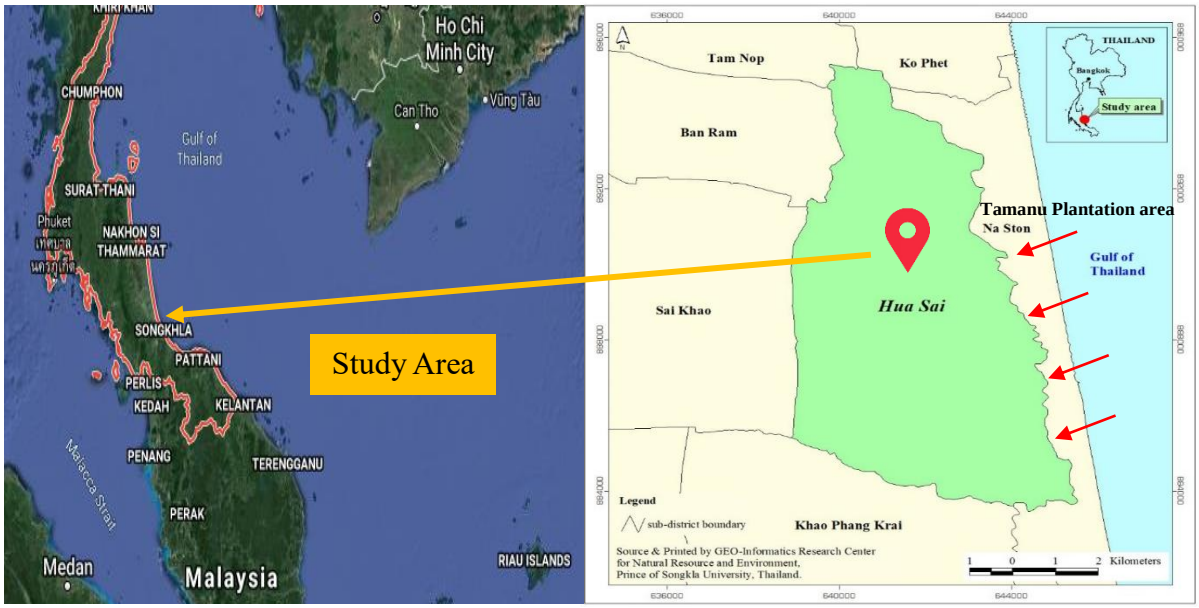


Figure 1 Maps showing the sources of tamanu kernels and coconut meat, in Hua Sai district, Nakhon Si Thammarat province, Thailand (GEO-Informatics Research Center for Natural Resource and Environment, Southern Regional Center of Geo-Informatics and Space Technology, 2019) [4].

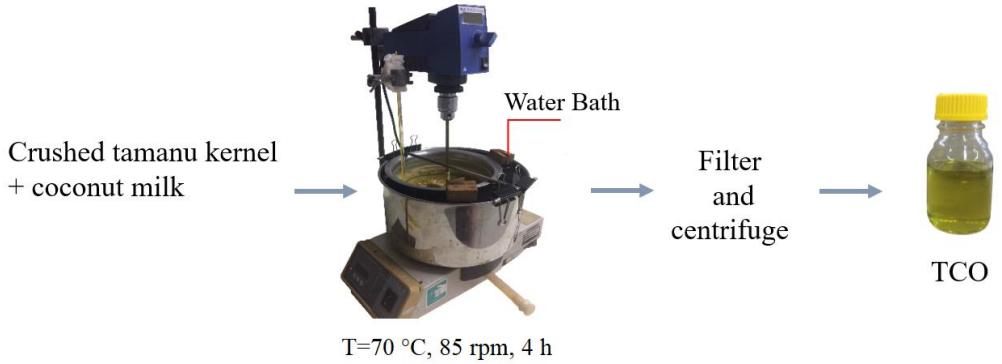


Figure 2 Steps in preparing tamanu and coconut oil mix (TCO)

collect the filtrate, coconut milk. The weight of added distilled water was equal to the weight of the grated coconut meat.

2.1.1 Coconut oil from thermal extraction

The coconut milk obtained from 1 kg of GCM was put in a stainless-steel pot equipped with straight blade stirrer, which was placed in a water bath (EYELA SB-651 Digital Water Bath). The temperature was set to 70 and 80 °C because overheating some ingredients (sugar, protein, and minerals) causes contamination of the oil [5], the stirrer speed was 85 rpm, and the duration of extraction was 5 h. The range 70 – 80 °C also matches the conditioning temperature for saponification reaction of fatty acid with lye [6]. When the water of coconut milk was mostly evaporated, coconut oil and bulky solid residuals remained in the pot. The solids were then removed by filtering and centrifuging at 5000 rpm, 20 min to obtain CO.

2.1.2 Tamanu oil from soxhlet extraction

The tamanu kernels were dehydrated at 40 °C, 8 h and crushed to approximately 1-2 mm particle size. These crushed tamanu kernels were placed in the thimble of a Soxhlet extractor, and extracted with n-hexane ('RCI Labscan' Hexane, AR Grade, Thailand) as the solvent for 6 h at 70 °C. Evaporating off the n-hexane using rotary evaporator, controlled at 37 °C [7].

2.1.3 Tamanu and coconut oil mix from thermal extraction

Oil extraction from tamanu kernels with coconut milk (co-extraction). The tamanu kernels were prepared for extraction of oil by crushing to 1-2 mm size. Then, thermal extraction was done using coconut milk as the solvent medium, and the weight ratio of tamanu kernel and grated coconut meat was set to 0.15, 0.25 or 0.35. The proper coconut milk mixed with crushed tamanu kernel was put in a stainless-steel pot equipped with straight blade stirrer that was placed in a water bath. During extraction the temperature was 70 °C to 80 °C, the stirrer speed was 85 rpm, and the duration of extraction was 4 h. Then, a filter cloth was used to separate extract from solid residues, followed by centrifugation at 5000 rpm for 20 min. Figure 2 summarizes the procedure for extracting tamanu and coconut oil mix (TCO). The obtained oil was kept in Duran bottles and put a refrigerator at 4 °C before analyses or soap processing.

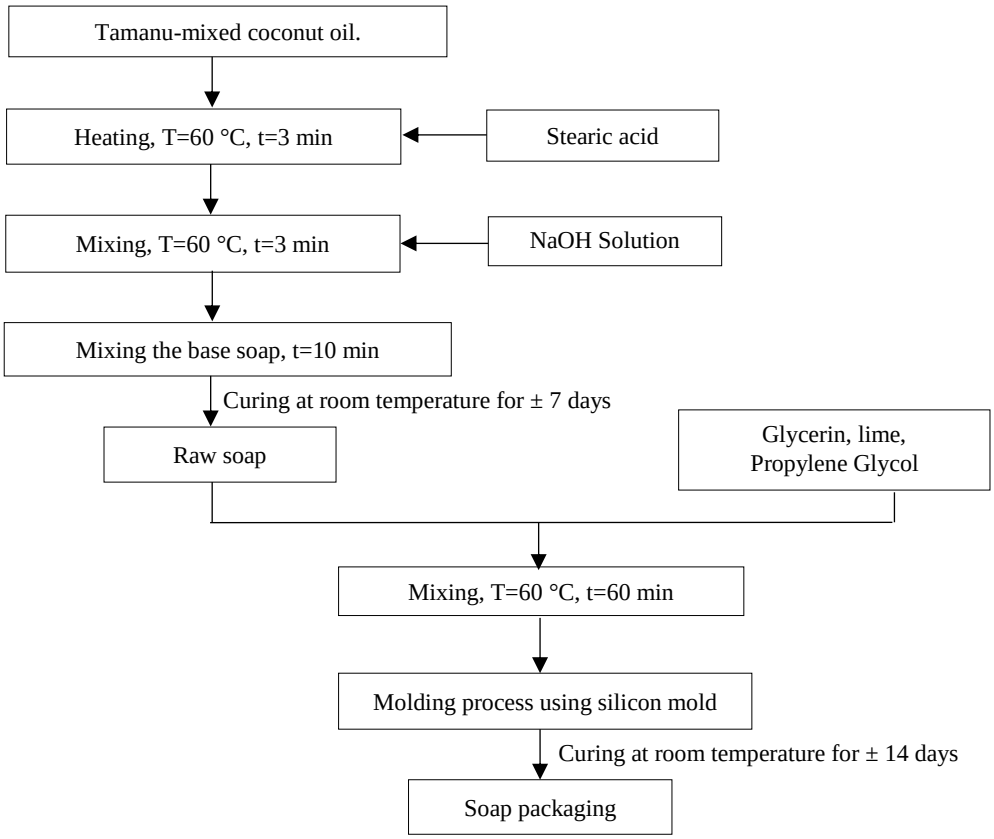


Figure 3 Flow diagram of the making soap process.

2.2 Oil quality analysis

2.2.1 Determination of saponification number (SN)

An AOAC 2000 method was used for determining the SN. In brief, 5 g of oil was placed in a round bottom flask for reflux, 50 ml of potassium hydroxide ethanolic solution was added, heating was done on a hot plate for 30 min, and the sample was allowed to cool down. The titration was then done with 0.5 N hydrochloric acid using phenolphthalein as indicator, so that at end point the solution turns from pink to colourless. Similar determination was done with a blank in which distilled water replaced the oil [8].

2.2.2 Determination of iodine value (IV)

Iodine value (AOAC 2000) was determined from a 0.4 g oil sample in a conical flask with 20 ml carbon tetrachloride added to dissolve the oil, followed by adding 25 ml of Wij's iodine solution. The flask was covered, shaken and placed in the dark for 50 min at room temperature. Then 20 ml of 10% potassium iodide solution and 200 ml of distilled water were added. The content was titrated with 0.1 N sodium thiosulphate until the yellow colour disappeared. About 2 ml of 1% starch indicator was added and the titration continued until the blue colour disappeared. The blank had no oil added [9].

2.2.3 Determination of acid value (AV)

Acid Value (AOAC 2000) was determined by accurately weighing an appropriate amount of the cooled oil sample in a 250 ml conical flask and adding 100 ml of freshly neutralized hot ethyl alcohol and about 1 ml of phenolphthalein indicator solution. The mixture was boiled for about 5 min and titrated while hot against 0.10 sodium hydroxide solution with vigorous shaking during the titration, until a permanent pink colour emerged [10].

2.2.4 Determination of fatty acid composition

The fatty acid composition was diluted with ethanol and estimated by gas chromatography (GC) with a GC-FID (Agilent 7890, USA) fitted with a capillary column (30 m length, 0.32 mm diameter, 0.25 μm film thickness) and initial temperature of 210 °C held for 12 min, then ramped at 20 °C/min to 250 °C and held for 8 min. Helium was used as the carrier gas of 10 mL/min and 50:1 split ratio. The injector and detector temperatures were set at 290 °C and 300 °C, respectively. The identification of FFA was referred to the mass spectra to those from the National Institute of Standards and Technology (NIST) libraries [11].

2.3 Soap making from CO and TCO

The soaps derived from CO and TCO were assessed for saponification numbers. The raw soap making was conducted at 60 °C in a water bath. The mixture of oils, stearic acid, and sodium hydroxide was quickly stirred for 10 min and poured to the mold, and was then allowed to cure for a week at room temperature. Also glycerin, propylene glycol, and lime juice were added to the raw soap at 60 °C for 60 min to obtain a homogeneous blend. The soap bars were cured for at least two weeks at room temperature after pouring to the mold size 100 g (width 5.8 cm, length 7 cm, height 2.5 cm). Figure 3 shows the soap making steps in a flow diagram.

Table 1 The analytical results of saponification (SN), iodine (IV) and acid (AV) for coconut (CO) and tamanu oils (TO).

Oil	SN (mgKOH/g)	IV (I_2/100g)	AV (mgKOH/g)	% Oil Yield
CO	258 ±2.645	10.97 ±0.208	0.60 ±0.01	18.3*
TO	194 ±2.645	84.03±0.551	15.07 ±0.305	31.6**

* calculated from g of oil/100g of GCM
** calculated from g of oil/100g of RTK

Table 2 The analytical results of SN, IV and AV for the mixed oils from tested ratios of RTK to GCM

Characteristics	RTM/GCM		
	0.15	0.25	0.35
SN (mgKOH/g)	245±1.322	230±0.541	210±0.210
IV (I_2/100g)	27.58±0.153	49.5±0.308	56.11±0.125
AV (mgKOH/g)	3.49±0.205	8.03±0.305	12.72±0.140
% Total oil yield*	14.7	16.1	18.0

* calculated from g of oil/100g of RTK and GCM

2.4 Soap quality analysis

2.4.1 Effectiveness in lathering and cleaning test

On considering the cleaning properties of the soap samples, oil droplets were dropped on four separate filter papers. The filter papers with oil droplets were soaked in separate test tubes containing soap solution (2 g soap / 100ml distilled water) shaking each tube vigorously for 1 min. The filter papers were removed and washed with distilled water and the cleanliness of each filter paper was assessed [12].

2.4.2 Moisture content

Moisture content (AOAC 2000) was determined by drying a 10 g sample to a constant weight at 105 ℃. It was allowed to cool and then reweighed [9].

2.4.3 Free caustic alkali

Free caustic alkali (ISO 456:1973) was determined by heating 200 g of ethanol in a round bottom flask for 5 min to remove carbon dioxide, then cooling it down to 70 ℃. Four drops of phenolphthalein indicator was added and titration with potassium hydroxide ethanolic solution was continued until the solution turned pink. A 5 g sample was weighted into stock solution and refluxed until the sample completely dissolved, stopped heating when the temperature dropped to 70 ℃, then titrated with hydrochloric acid until the pink color disappeared [13].

2.4.4 Ethanol insoluble material

Material insoluble in ethanol (AOCS) was determined from a 5 g soap sample dissolved in 50 ml hot ethanol and quantitatively captured on a pre-weighed filter paper. The residue was dried in the oven at 105 ℃ for 30 min, cooled and weighed again [14].

2.4.5 Total fatty matter

Total fatty matter (ISO 685:1975) was determined by mixing a certain amount of the soap and hot water in separating funnels. A few drops of the methyl orange and hydrochloric acid solutions were added. The funnels were shaken vigorously and cooled until the contents inside reached the ambient temperature. Petroleum ether as a hydrophobic solvent was added, continue shaking until the aqueous layer has become clear. The aqueous phase was poured out and the petroleum ether was evaporated in the water bath. Ethanol as a universal solvent and a few drops of phenolphthalein was added, the obtained solution was titrated with the potassium hydroxide solution, the titrant volume of potassium hydroxide was recorded. The ethanolic potassium hydroxide solution was evaporated by adding acetone via the water bath. Acetone was evaporated in a controlled water bath at a temperature of 103 ºC for 15 min. The remaining matter in a desiccator was cooled and weighed [13].

2.4.6 pH

The pH was determined from 1 g of soap dissolved in 100 ml distilled water and shaken until homogeneous, then stored for 2 h. The pH was determined with a pH meter (models PH100 and PH110) [15].

2.4.7 Foam stability

A 4 g sample of soap was dissolved in 100 ml of distilled water, stirring until well mixed and poured into a 200 ml measuring cylinder, then shaken vigorously for 4 min. It was allowed to stand for 15 to 20 min. The time taken for the foam to collapse was determined using a stopwatch [16].

3. Results and discussion

The analyzed oil characteristics were SN, IV, and AV together with oil yield, shown in Table 1 for raw tamanu kernel (RTK) and grated coconut meat (GCM). The tamanu and coconut oils were obtained by Soxhlet and thermal extraction, respectively. Table 2 shows similar characteristics of the mixed oil from co-extraction along with oil yields. Furthermore, Figure 4 shows appearances of the oils and the solid residues.

Table 3 and Table 4, show the free fatty acid compositions in both CO and TO and those in TCO respectively. The tables also include proportions of unsaturated and saturated oils. The oil yields from crushed tamanu mixed with coconut milk have been calculated based on TO and CO oil yields. Table 4 also shows the extraction degrees of TO which are higher than those of CO. The comparative degrees affect the FFA content in the whole raw material. For a clear explanation of the re-adsorbed amount of oil in solid residue, Figures 5 and 6 enable comparing percentage of FFA between the separate extracts and co-extract from RTK and GCM in similar proportions. Table 5

Figure 4 Appearances of CO, TO and TCO (a); and CO and TCO solid residues (b).

Table 3 FFA compositions in CO and TO

FFA			FFA % (w/w)	
			CO	**TO**
Unsaturated fatty acid (UFAs)	Oleic	C18:1	6.47±0.54	36.77±0.35
	Linoleic	C18:2	2.10±0.35	30.83±0.59
	Linolenic	C18:3	0	0.62±0.11
Saturated fatty acid (SFAs)	Caprylic	C8:0	6.20±0.23	0
	Capric	C10:0	6.50±0.19	0
	Lauric	C12:0	48.90±1.02	0
	Myristic	C14:0	17.73±0.10	0.02±0.08
	Palmitic	C16:0	9.10±0.18	8.20±0.03
	Stearic	C18:0	2.90±0.27	13.25±0.20
	Arachidic	C20:0	0	0.02±0.21
	Behenic	C21:0	0	5.06±0.13
	Lignoceric	C24:0	0	5.23±0.03
% UFAs			8.7	68.2
% SFAs			91.3	31.8

Table 4 FFA compositions in RTK/GCM blends at ratios 0.15, 0.25 and 0.35

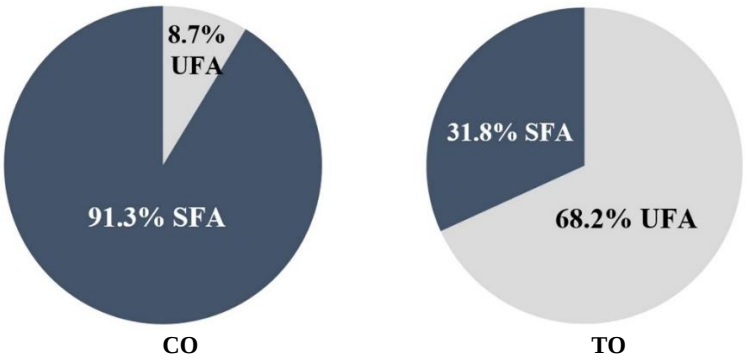
FFA			FFA of RTK/GCM, %(w/w)		
			0.15	**0.25**	**0.35**
Unsaturated fatty acid (UFAs)	Oleic	C18:1	17.35±0.13	22.71±0.05	28.34±0.12
	Linoleic	C18:2	12.41±0.02	17.58±0.24	22.78±0.08
	Linolenic	C18:3	0.22±0.23	0.34±0.15	0.47±0.18
Saturated fatty acid (SFAs)	Caprylic	C8:0	3.96±0.11	2.85±0.23	1.73±0.15
	Capric	C10:0	4.17±0.14	2.95±0.04	1.63±0.08
	Lauric	C12:0	31.35±0.04	22.56±0.02	13.31±0.05
	Myristic	C14:0	11.38±0.08	8.19±0.01	4.45±0.23
	Palmitic	C16:0	8.78±0.07	8.63±0.14	8.45±0.02
	Stearic	C18:0	6.61±0.03	8.46±0.31	11.01±0.14
	Arachidic	C20:0	0.01±0.15	0.01±0.02	0.01±0.11
	Behenic	C21:0	1.85±0.11	2.73±0.13	3.82±0.03
	Lignoceric	C24:0	1.87±0.03	2.85±0.02	3.96±0.02
% UFAs			30.0(18*)	40.7(24*)	51.6(30*)
% SFAs			70.0(82**)	59.3(76**)	48.4(70**)

* Percentage of UFAs based on oil from separated extraction of RTK and GCM
** Percentage of SFAs based on oil from separated extraction of RTK and GCM

displays the oil extraction efficiency by proportions of RTK and GCM. The results also confirm the adsorption of several FFA onto the solid residue.

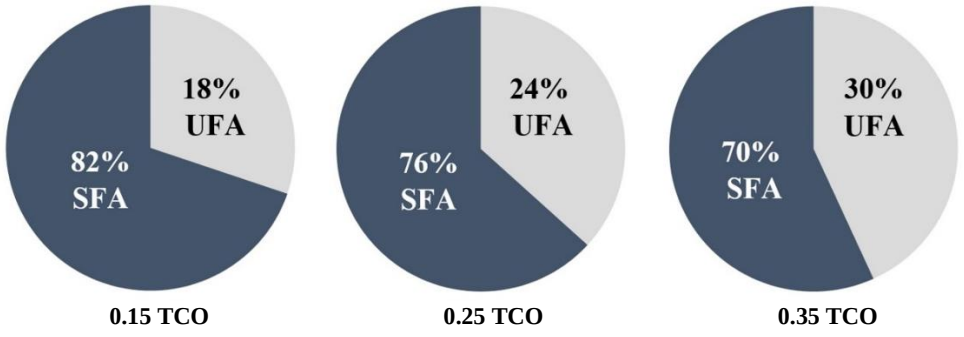
The soaps obtained from CO and TCO were subjected to physical and chemical analyses with results shown in Table 6 and 7. Figure 7 shows color photos of both soaps. The results indicate that the soaps have different colors are the oils used to prepare them. The surface texture of the soap obtained from CO was harder than that from TCO, which is related to the level of IV in the oil to make the soap because the high IV oil always provides soap of high TFM and high moisture content that provide the softer surface texture [12].

Regarding lathering ability and cleansing, the CO soap provided a large amount of bubbles and the cleansing was better than with TCO soap. The amounts of lauric acid and myristic acid play an important role in these properties because these FFA relate to foaming and cleansing ability of the soap [17].

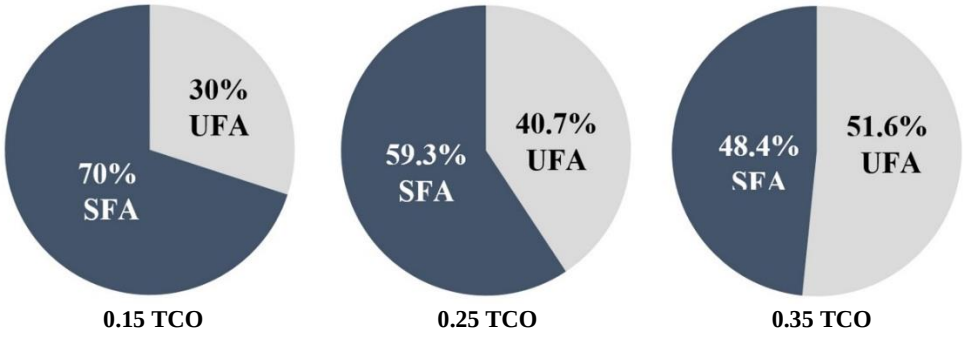


(a) (b)

Figure 5 The comparison percentage of UFAs and SFAs between the CO (a) and TO (b).



(a)



(b)

Figure 6 the comparison between the percentage of FFA between separate extracts (a) and co-extracts (b) with similar proportions of RTK and GCM.

Table 5 The oil extraction efficiency for the tested RTK: GCM mixes

Characteristics	RTK: GCM (Total weight = 1000 g)		
	0.15:0.85	0.25:0.75	0.35:0.65
Obtained TCO (g)	146.95	161.35	179.59
Oil from separated extraction (g) *	202.95	216.25	229.55
% Oil extraction efficiency	72.41	74.61	78.23

* Calculated based on the data in Table 1

Table 7 shows that the moisture content in TCO soap was higher than in CO soap, which led to less foaming and poorer cleansing. Furthermore, a high moisture content in soap shortens its shelf life [18]. Ethanol insoluble material in a soap indicates the level of contamination caused by the alkaline used in production of the soap [19]. The results reveal that both CO soap and TCO soap satisfied the standard regarding alkalinity contamination. Free caustic alkali prevents a soap from becoming oily [14] and is retained from improper or incomplete saponification [18]. The free caustic alkali value of soap should be less than 0.05%. The results show that NaOH could almost completely react with both CO and TCO since no free caustic alkali was detected.

The TFM indicates the amount of oil contained in the soap, which should be more than 76.5%, and it describes the extent of saponification reactions. The TFM also relates to

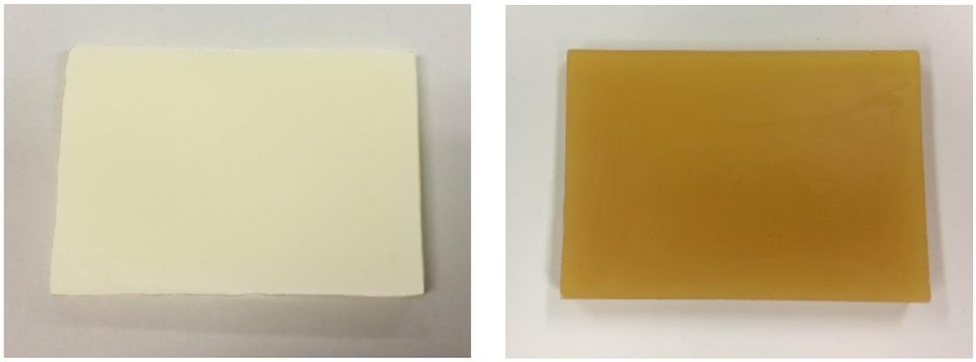
Table 6 Color, texture, lathering and cleaning properties of the soaps, and their typification based on these characteristics

Fat/oil	Color of soap	Texture	Lathering	Cleaning	Type of soap
CO	White	Hard	Vary Good	More effective	Toilet soap
TCO	Yellow	Soft	Fairly Good	Effective	Beauty soap

Table 7 Chemical components in the soaps

Component	CO Soap	TCO Soap	Standard*
Moisture content (%)	8.13±0.208	8.23±0.115	-
Insoluble material in ethanol (%)	0.77±0.015	0.76±0.005	<2.5
Free caustic alkali (%)	Not found	Not found	<0.05
Total fatty matter (%)	79.73±0.208	83.86±1.594	> 76.5
pH	9.46±0.057	8.73±0.057	8-10
Foam stability (min)	3.70±0.200	2.85±0.050	-

* TCPS 94-2546 and TIS 29-2545 Standard



(a) (b)

Figure 7 Coconut oil soap (a), and TCO soap (b).

the moisture content and the amount of fat in the soap remaining after saponification. However, dry skin needs a soap that has a high TFM of about 80%, which helps to replenish the skin moisture. Besides, a high oil content in soap leaves a coating on the skin for a day [16]. Thus, TCO soap is better than CO soap as regards skincare. pH is an important parameter showing whether a soap is alkaline or acidic, and too alkaline soap may irritate the skin [20] again the TCO soap should be more suitable for skin than the CO soap.

Foam stability results show that the soap from CO gave more stable foam than the TCO soap, probably caused by palmitic acid and myristic acid fractions in the soaps [6]. More palmitic and myristic acid induced lower moisture content in the soap, and the strength of foam is inversely related to soap moisture content [18].

4. Conclusions

Co-extraction of tamanu oil with coconut milk was conducted at atmospheric pressure by thermal extraction at 70 °C. Some fatty acids obtained from coconut oil were likely to adsorb onto the mixture of crushed tamanu kernel and solid residues from coconut meat. The yields of TCO were in the range from 14 to 18% depending on the RTK/GCM ratio. A suitable ratio for soap production having unsaturated and saturated fatty acids in 40/60 proportions had RTK/GCM=0.25, with a total oil yield and oil extraction efficiency of 16% and 75%, respectively. The soaps made from CO and TCO were analysed according to TCPS 94-2546 and TIS 29-2545 standards, and the TCO soap was superior to the CO soap in many aspects. The solid residues from extraction can be collected to make other products, for example by mixing with herbs to make herbal balls for spa use, or in mixes with other skincare products. This co-extraction for soap making would be suitable for rural beach areas in the Pacific islands, South and Southeast Asia, and Africa, with abundant coconut and tamanu trees.

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