

Physical Refining of Coconut Oil: Effect of Crude Oil Quality and Deodorization Conditions on Neutral Oil Loss

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ABSTRACT: In the present study, neutral oil loss (distillative and mechanical carry-over) during physical refining of coconut oil was quantified. Neutral oil loss seems to depend on both the crude oil quality and the process conditions during deodorization. The distillation of volatile glyceridic components (mono- and diglycerides), originally present in the crude oil, was confirmed as the major cause for the neutral oil loss. The amount of these volatile components in crude coconut oils cannot be derived as such from the initial free fatty acid content. A lower deodorization pressure with less sparge steam resulted in a larger neutral oil loss than a higher pressure with more steam. A "deodorizability" test on a laboratory scale under standardized conditions (temperature = 230°C, pressure = 3 mbar, time = 60 min, sparge steam = 1%), to evaluate crude oil quality and to obtain a more accurate prediction of the expected neutral oil loss and free fatty acid content in the fatty acid distillate, is described.

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KEY WORDS: Coconut oil, deodorization, free fatty acids, neutral oil loss, volatile components.

Coconut oil belongs to the group of vegetable oils named lauric oils, which are characterized by a high level of short-chain fatty acids (>50% C6:0–C12:0) and low unsaturation (Table 1) (1–3). Other known lauric oils are palm kernel oil, babassu, cohune, and cuphea (4).

Crude coconut oil, as well as palm kernel oil, can have a relatively high free fatty acid (FFA) content (between 1 and 6%) due to enzymatic hydrolysis prior to extraction/preparation (5–7). High FFA content implies the presence of mono- and diglycerides. On the basis of an earlier study by Loncin (8), it can be estimated that a bleached coconut oil with 3% FFA contains about 3% diglycerides and about 1% monoglycerides. Mono- and diglycerides can be analyzed by capillary gas–liquid chromatography as described in the AOCS Official Method Cd 11b-91 (9). However, for oils and fats with short- and long-chain fatty acids like coconut oil and palm kernel oil, accurate results are difficult to obtain owing to partial peak overlap. Alternatively, α -monoglycerides can also

be detected titrimetrically according to the AOCS Official Method Cd 11-57 (9).

By assuming a random hydrolysis, the monoglycerides in coconut oil can contain up to 50% short-chain fatty acids (C6–C12). These components have a volatility similar to the long-chain fatty acids (C16–C18), e.g., the boiling points of glycerol monolaureate and stearic acid at 1.33 mbar are 186 and 183°C, respectively (10). In chemical refining, monoglycerides, and to a lesser extent diglycerides, are predominantly removed during neutralization because of their relatively higher affinity for water. However, coconut oil is rarely chemically refined. Because of the high initial FFA content, physical refining is mostly preferred for lauric oils (11–13).

In the physical refining of lauric oils, the FFA content of the obtained fatty acid distillate (FAD) is always lower than the FFA level in distillates from physically refined oils rich in long-chain fatty acids (e.g., palm, corn, and rapeseed oils). Accordingly, the calculated neutral oil losses in lauric oils distillates are considerably higher.

In the present study, the distillation of volatile mono- and diglycerides was confirmed as the major neutral oil loss during physical refining of coconut oil. Neutral oil losses seem to depend on both the crude oil quality and the process conditions during deodorization. A laboratory scale "deodorizability test" is described to evaluate crude oil quality.

EXPERIMENTAL PROCEDURES

Materials. Seven bleached coconut oil samples from different origins were obtained and stored in the dark at 4°C until analysis. Fully refined soybean oil obtained from the local supermarket was taken as a reference sample. Physical refining experiments were conducted in a laboratory-scale batch deodorizer (Scheme 1). The batch size was 250 g of oil.

All solvents and reagents were of analytical grade and purchased from Acros Organics (Geel, Belgium).

Analytical procedures. The FFA content was determined according to the AOCS Official Method Ca 5a-40 (9). Fatty acid methyl esters were prepared and subsequently analyzed with a Hewlett-Packard 5890 gas–liquid chromatograph, equipped with a flame-ionization detector and on-column injector (Hewlett-Packard, Brussels, Belgium) as described in the AOCS Official Methods Ce 1-62 and Ce 2-66 (9). A

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TABLE 1
Fatty Acid Composition (wt %) of Coconut Oil Samples of Different Origin

Fatty acid	Oil origin							Mean
	CNO-1	CNO-2	CNO-3	CNO-4	CNO-5	CNO-6	CNO-7	
C6:0	0.7	0.7	0.7	0.4	0.5	0.7	0.5	0.6
C8:0	7.3	7.3	7.1	6.7	7.3	8.0	7.3	7.3
C10:0	5.8	5.8	5.8	5.7	5.9	6.1	6.2	5.9
C12:0	46.0	46.6	46.0	47.4	46.6	45.8	47.7	46.6
C14:0	18.9	18.8	18.4	18.7	18.8	18.4	18.0	18.6
C16:0	9.4	9.4	9.8	9.5	9.5	9.6	9.2	9.5
C18:0	2.9	2.9	3.1	2.8	2.9	2.6	1.6	2.7
C18:1	7.1	6.8	7.3	7.2	6.8	6.9	6.7	7.0
C18:2	1.9	1.7	1.8	1.6	1.7	1.9	2.8	1.9
MMW ^a	214.0	213.6	214.5	214.4	213.8	213.1	213.0	213.8

^aMMW, mean molecular weight (g).

fused-silica capillary column, coated with 100% cyanopropyl polysiloxane (CP-Sil 88, 50 m × 0.25 mm internal diameter; 0.20 μm film thickness; Chrompack, Middelburg, The Netherlands), was used with hydrogen as carrier gas. Initial oven temperature was set at 60°C and increased at a rate of 5°C/min to 200°C, where it was held until completion of the analysis. Quantitative analyses were performed with a HP ChemStation.

RESULTS AND DISCUSSION

The fatty acid composition of the different bleached coconut oil samples showed only small variations (Table 1). The amount of short-chain fatty acids (C6:0–C12:0) varied from 59.6 to 61.7%. For the analyzed coconut oils, the calculated mean molecular weight (MMW) of the fatty acids ranged from 213.0 to 214.5 g (Table 1). The FFA content of lauric oils is generally expressed as % lauric acid (C12:0, MW = 200 g). A more correct way would be to express the FFA content in terms of MMW:

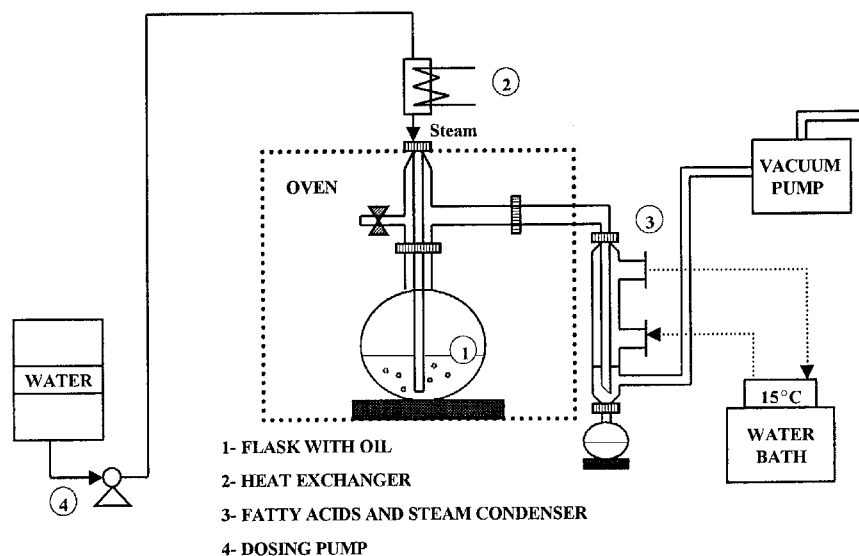
$$\text{FFA}_{\text{MMW}} = \text{FFA}_{\text{C12:0}} \frac{\text{MMW}}{\text{MW}_{\text{C12:0}}} (\%) \quad [1]$$

where $\text{FFA}_{\text{C12:0}}$ and FFA_{MMW} are the FFA content expressed as % lauric acid and as % MMW, MMW is the calculated mean molecular weight of fatty acids (g), and $\text{MW}_{\text{C12:0}}$ is the molecular weight of lauric acid ($\text{MW}_{\text{C12:0}} = 200 \text{ g}$).

This would result not only in a slightly higher FFA level for the crude and refined coconut oil but also in a considerably higher FFA level in the FAD. The latter conclusion is especially important when the neutral oil loss during deodorization/physical deacidification is considered. In general, this neutral oil loss is calculated from the FAD and expressed by the following equation:

$$\text{neutral oil loss} = \frac{\text{FAD flow} (100 - \text{FFA}_{\text{FAD}} - \text{unsaps})}{100} (\%) \quad [2]$$

$$\text{FAD flow} = \frac{100 (\text{FFA}_{\text{OIL IN}} - \text{FFA}_{\text{OIL OUT}})}{\text{FFA}_{\text{FAD}} - \text{FFA}_{\text{OIL OUT}}} (\%) \quad [3]$$



SCHEME 1

where $FFA_{OIL\ IN}$, $FFA_{OIL\ OUT}$, and FFA_{FAD} are the FFA content of the crude oil, refined oil, and FAD (%), respectively, and “unsaps” is the unsaponifiable matter (%). The amount of unsaponifiable matter in coconut oil is very low and therefore is not taken into account in the calculation.

In practice, neutral oil losses are usually somewhat higher due to side reactions (e.g., hydrolysis, polymerization) (14,15), which are difficult to quantify.

Neutral oil loss will be overestimated when, in case of coconut oil, the FFA in the distillate is expressed as lauric acid (Table 2). Therefore, in our study the FFA content was expressed on an MMW basis to approach as much as possible the real neutral oil loss.

A bleached coconut oil sample with an initial FFA content of 3.40% (MMW = 213.8) was deodorized in the lab-scale deodorizer (Scheme 1) under different conditions. As expected, the FFA level of the refined oil ($FFA_{OIL\ OUT}$) decreased from 0.251 to 0.075 to 0.020% with increasing temperature from 190 to 210 to 230°C, respectively (Table 2). A similar effect of the process temperature on the FFA content of the distillate (FFA_{FAD}) was observed. For a same stripping efficiency of FFA ($FFA_{OIL\ OUT}$ below 0.03%), the FFA level of the FAD increased (and hence neutral oil loss in the FAD decreased) with increasing pressure and increasing sparge steam (83.10 vs. 76.23% FFA in the FAD; see Table 2, Experiments 6 and 3). Today, oil refineries operate at lower deodorization pressure in order to reduce the amount of sparge steam and, consequently, to decrease the motive steam consumption of the vacuum unit as well as the carry-over. However, from the results of our study it can be concluded that a combination of a low pressure and a low amount of sparge steam is not recommended for the physical refining of lauric oils, as a much higher neutral oil loss is obtained. A possible explanation for the apparent relationship between $FFA_{OIL\ OUT}$ and FFA_{FAD} , and hence high neutral oil loss in the FAD with decreasing pressure, could be the earlier-mentioned similar volatility of long-chain fatty acids (C16–C18) and short-chain monoglycerides (C6–C12). As a consequence, operating conditions necessary to achieve a low residual FFA level in the refined oil would also result in the distillation of short-chain monoglycerides (especially at a lower deodorization pressure)

leading to a lower FFA level and hence higher neutral oil loss in the FAD. Based on the obtained data, it is therefore recommended during physical refining of coconut oil to operate at a higher pressure (e.g., 3 mbar), a higher sparge steam (e.g., 1%) and a temperature of, for example, 230°C.

A series of laboratory-scale deodorization trials was set up to illustrate the effect of sparge steam and temperature on FFA and neutral oil loss in the FAD. A bleached coconut oil with an initial FFA content of 5.15% (MMW = 213.0) was deodorized for 60 min at two different temperatures (190 and 230°C), a pressure of 3 mbar, and with addition of 1% sparge steam. Afterward, the deodorized oil was redeodorized several times under the above-mentioned conditions. The fatty acid composition of the FAD, collected during high sparge steam/low-temperature deodorization, indicated a selective distillation of volatile components (Table 3). Sparge steam at 1.2% resulted mainly in distillation of short-chain fatty acids (63.7% C6:0–C12:0 fatty acids in FAD). By increasing the amount of sparge steam to 3.6%, long-chain fatty acids (C16–C18) were distilled. As a consequence, the MMW of the FAD increased from 210.4 to 250.4–255.1. By a further increase of sparge steam (5–7%), short-chain mono- and diglycerides (C6–C12) were removed. As a result, the MMW of FAD decreased from 255.1 to 231.7. The neutral oil loss, however, remained in all cases very low (below 0.1% each time). To achieve the residual FFA level in the refined oil below 0.05%, about 1–2% more sparge steam is required. In practice, such high sparge steam consumption (8–9%) is not economic. At 230°C with 1.1% of sparge steam a more efficient distillation of volatile components resulted. As a consequence, short-chain monoglycerides and to a lesser extent diglycerides were distilled together with fatty acids, resulting in a lower FFA in the refined oil (below 0.03%) and a higher neutral oil loss in the FAD (0.97%).

In general, the neutral oil loss in the FAD can be divided in a distillative loss and a loss due to mechanical carry-over. The latter loss includes mainly triglycerides and is to a certain extent related to the deodorizer design (14). The distillative loss mainly consists of volatile components such as flavors, contaminants (pesticides, light polycyclic hydrocarbons), unsaponifiable matter (tocopherols, sterols) and

TABLE 2
Effect of Deodorization Parameters on Neutral Oil Loss for a Given Quality Coconut Oil^a

Exp. no.	Process conditions				$FFA_{OIL\ OUT}$ ^b		FFA_{FAD} ^b		Neutral oil loss	
	Temp. (°C)	Steam (%)	Pressure (mbar)	Time (min)	(%C12:0)	(%MMW)	(%C12:0)	(%MMW)	(%C12:0)	(%MMW)
1	190	0.6	1.6	60	0.235	0.251	91.20	97.40	0.28	0.08
2	210	0.8	1.6	60	0.070	0.075	84.63	92.16	0.57	0.28
3	230	0.7	1.6	60	0.019	0.020	71.21	76.23	1.28	1.05
4	230	0.6	2.3	60	0.033	0.035	76.23	81.91	0.98	0.74
5	230	0.6	3.0	60	0.035	0.037	78.00	83.50	0.89	0.66
6	230	1.2	3.0	60	0.017	0.018	77.20	83.10	0.94	0.69

^aInitial free fatty acid (FFA) content = 3.40%, MMW = 213.8.

^b $FFA_{OIL\ OUT}$, FFA content of refined oil; FFA_{FAD} , FFA content of fatty acid distillate. FFA content is expressed on lauric acid (%C12:0) and on mean molecular weight (%MMW) basis.

TABLE 3
Effect of Sparge Steam and Temperature on FFA and Neutral Oil Loss in the Distillate for a Given Quality Coconut Oil^a

	Sparge steam at 190°C (%)						Sparge steam at 230°C (%)		
	1.2	2.4	3.6	4.8	6.0	7.2	1.1	2.2	3.3
FFA _{OIL OUT} (%MMW)	0.556	0.304	0.197	0.127	0.083	0.063	0.027	0.011	0.005
FFA _{FAD} (%MMW)	98.07	75.17	64.32	48.48	35.65	24.87	84.08	5.63	2.72
MMW _{FAD} (g)	210.4	250.4	255.1	247.4	237.2	231.7	214.2	197.2	195.1
C6:0–C12:0 fatty acids (wt%) ^b	63.7	14.3	17.8	28.2	39.1	44.3	59.8	78.7	81.4
FAD flow (%)	4.71	0.34	0.17	0.14	0.12	0.08	6.09	0.28	0.22
Neutral oil loss ^c (%)	0.09	0.08	0.06	0.07	0.08	0.06	0.97	0.27	0.21
Total neutral oil loss (%)	0.44						1.45		

^aInitial free fatty acid content = 5.15%, MMW = 213.0, deodorization pressure = 3 mbar.

^bIn fatty acid distillate (FAD).

^cNeutral oil loss is calculated on FFA levels expressed as %MMW. See Table 2 for abbreviations.

also, in the case of lauric oils, volatile mono- and diglycerides.

During physical refining of lauric oils, the contribution of the distillative loss to the neutral oil loss may be high owing to the presence of volatile short-chain mono- and diglycerides in the crude oil.

A well-defined series of experiments was conducted to confirm this hypothesis. Two fully refined oils (physically refined coconut oil and chemically refined soybean oil) were re-deodorized for 60 min in the laboratory-scale deodorizer at two different temperatures (190 and 230°C), a pressure of 3 mbar, and with addition of 1.1% sparge steam.

At 190°C, the FAD flow was low and almost equal for both oils (0.06–0.07%) (Table 4). FFA levels before and after deodorization were nearly the same, which illustrated that no distillation occurred under these process conditions. Therefore, the FAD flow can be considered as a loss due to mechanical carry-over (liquid entrainment in the vapor phase). At 230°C, FFA were distilled from both oils resulting in an increased FAD flow. Nevertheless, the neutral oil loss, calculated according to Equation 2, remained at the same level for

soybean oil (0.08%). Consequently, the neutral oil loss during deodorization of soybean oil is almost exclusively due to mechanical carry-over. For soybean oil, the distillative loss is very low because volatile mono- and diglycerides are only present in negligible amounts. This is not the case for coconut oil. Redeodorization at 230°C increased the neutral oil loss to about 0.52%, consisting mainly of a distillative loss. According to Carlson (14), losses due to mechanical carry-over are generally low and independent of the type of oil. Therefore, it can be assumed that the loss due to mechanical carry-over will be similar for soybean oil and coconut oil (about 0.1%). The remaining loss during deodorization of coconut oil at 230°C (about 0.42%) has to be considered as a distillative loss. Apparently, even in refined coconut oil, there are still volatile mono- and diglycerides that are distilled during redeodorization, thus increasing the neutral oil loss. The applied deodorization conditions had no significant effect on oil hydrolysis. “Extra produced” FFA varied between 0.015–0.020%.

Obviously, the distillative loss during physical refining largely depends on the amount of volatile components in the crude oil. Theoretically, this amount is correlated with the initial FFA content of the crude oil (15). Because of this relationship, neutral oil losses are estimated from the initial FFA content. It is generally assumed that the neutral oil loss will increase with increasing FFA content of the crude oil (14). A series of trials was conducted to find out if the initial FFA content could serve as a reliable indicator for the prediction of the neutral oil loss. Seven bleached coconut oils from different origins with an initial FFA content between 2.54 and 5.15% were deodorized on laboratory-scale under standardized conditions (temperature = 230°C, pressure = 3 mbar, time = 60 min, sparge steam = 1.1%). The neutral oil losses and FFA levels of the distillates are mentioned in Table 5. The loss due to mechanical carry-over was considered equal for all samples (about 0.1%) because of the identical deodorizer design and deodorization conditions. Based on the previous experiment, it may be concluded that the remaining loss, varying between 0.59 and 1.65%, is due to the distillation of volatile components (Table 5). No direct relationship could

TABLE 4
Determination of the Mechanical Carry-over and Distillative Losses for Coconut Oil and Soybean Oil^a

	RBD ^b coconut oil		NBD ^b soybean oil	
	Exp. 1 190°C	Exp. 2 230°C	Exp. 3 190°C	Exp. 4 230°C
FFA _{OIL IN} (%)	0.012	0.012	0.063	0.063
FFA _{OIL OUT} (%)	0.013	0.007	0.050	0.010
FFA _{FAD} (%)	ND	4.5	ND	45.1
Neutral oil losses				
FAD flow (%)	0.07	0.55	0.06	0.15
Distillative loss (%)	—	0.46	—	0.075
Hydrolysis (%)	ND	0.020	ND	0.015

^aPressure = 3 mbar, time = 60 min, steam = 1.1%.

^bRBD, refined, bleached, deodorized; NBD, neutralized, bleached, deodorized; FFA expressed on lauric acid basis for coconut oil and on oleic acid basis for soybean oil (%); ND, not detectable; distillative loss = FAD flow – FFA(hydrolysis) – FAD flow at 190°C (%); hydrolysis = FFA_{FAD} × FAD flow/100 – (FFA_{OIL IN} – FFA_{OIL OUT}) (%). Other abbreviations as in Table 2.

TABLE 5
Relationship Between Initial FFA Content of Coconut Oil and Neutral Oil Loss
During Laboratory-Scale Deodorization^a

	Oil origin						
	CNO-1	CNO-2	CNO-3	CNO-4	CNO-5	CNO-6	CNO-7
FFA _{OIL IN} (%MMW)	2.54	2.80	2.83	3.11	3.40	3.96	5.15
FFA _{OIL OUT} (%MMW)	0.019	0.019	0.019	0.011	0.018	0.040	0.027
MMW _{OIL} (g)	214.0	213.6	214.5	214.4	213.8	213.1	213.0
FFA _{FAD} (%MMW)	70.8	74.1	70.3	64.0	83.1	74.6	83.6
MMW _{FAD} (g)	215.2	214.7	214.1	211.2	215.3	213.0	213.3
Neutral oil loss ^b (%)	1.04	0.97	1.19	1.75	0.69	1.33	1.00

^aTemperature = 230°C, steam = 1.1%, pressure = 3 mbar, time = 60 min. Abbreviations as in Table 2.

^bNeutral oil loss is calculated on FFA levels expressed as %MMW.

be observed between the initial FFA content and the neutral oil loss. Some bleached oil samples (e.g., CNO-2 and CNO-5) apparently contained more volatile components than expected from the initial FFA content. These higher levels of volatile components may have a different origin. One possible explanation could be that FAD was added to these crude oils to increase its initial FFA content. For example, the initial FFA content of a crude coconut oil can be increased from 1.5 to 2.5% by addition of only 1.4% FAD with an FFA content of 70%. In this way, the amount of volatile components other than FFA is increased by 0.4%. These components will be distilled again during deodorization and consequently will result in a 0.4% extra neutral oil loss. In practice, this addition of volatile components to the crude oil cannot be detected with the existing analytical techniques.

Storage conditions of coconut kernels could be another possible explanation, as a number of enzymatic reactions can take place, resulting in a higher than theoretical amount of short-chain monoglycerides.

Figure 1 shows no direct relationship between the neutral oil loss and the initial FFA content of coconut oil of different origins (lines of calculated neutral oil loss were plotted according to Equations 2 and 3, assuming FFA_{OIL OUT} = 0.05%). For example, the initial FFA contents for CNO-4 and CNO-5 were 3.11 and 3.40%, respectively. Nevertheless, the neutral oil losses were 1.75 and 0.69%, respectively. Therefore, a more correct way would be to express the neutral oil loss on a relative basis, taking into account only the FFA of the FAD:

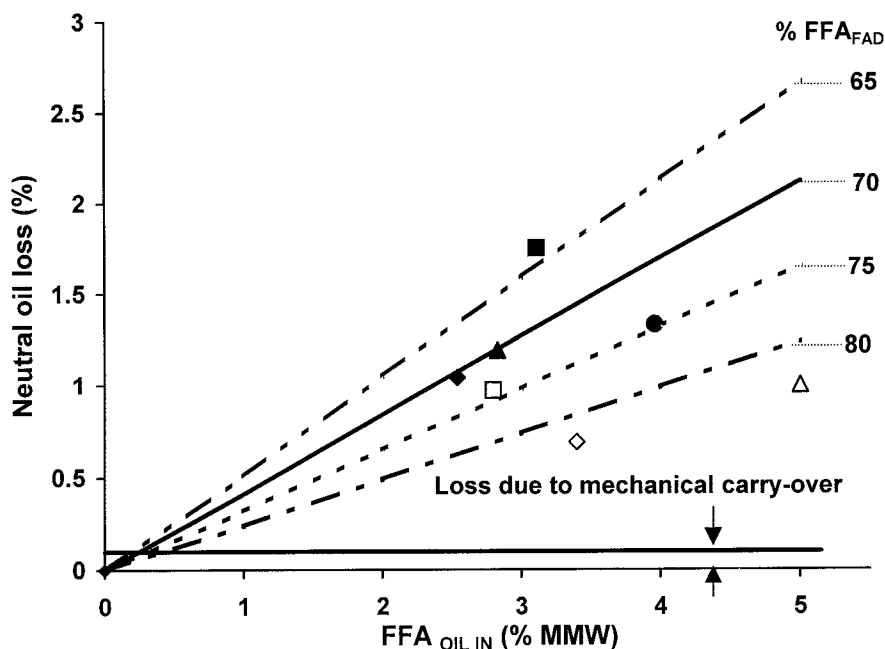


FIG. 1. Neutral oil loss (distillative and mechanical carry-over) during deodorization of coconut oil as a function of oil origin. Code: sample CNO-1 (◆); CNO-2 (□); CNO-3 (▲); CNO-4 (■); CNO-5 (◇); CNO-6 (●); and CNO-7 (△); “deodorizability test”: temperature = 230°C, pressure = 3 mbar, time = 60 min, sparge steam = 1%. FFA_{OIL IN}, free fatty acid content of crude oil; FFA_{FAD}, free fatty acid content of the fatty acid distillate; MMW, calculated mean molecular weight of fatty acids.

TABLE 6
Effect of FFA Content of Crude Coconut Oil and Fatty Acid Distillate on Profit Loss^a

FFA _{OIL IN} (%MMW)	FFA _{OIL OUT} (%MMW)	FFA _{FAD} (%MMW)	FAD flow ^b (%)	Neutral oil loss ^c (%)	Money from refined oil and FAD (U.S. \$/ton)	Profit loss ^d (U.S. \$/ton)
3	0.05	70	4.22	1.27	731.0	2.4
3	0.05	80 (reference)	3.69	0.74	733.4	
5	0.05	70	7.10	2.12	718.0	4.1
5	0.05	80 (reference)	6.19	1.24	722.1	

^aCalculated on plant capacity = 100 ton/d, price of refined oil = 750 U.S. \$/ton, price of fatty acid distillate = 300 U.S. \$/ton.

^bFAD flow was calculated according to the Equation 3.

^cNeutral oil loss was calculated according to the Equation 2.

^dProfit loss was calculated from Equation 5. For abbreviations see Table 2.

$$\text{relative neutral oil loss} = 100 - \text{FFA}_{\text{FAD}} - \text{unsaps} (\%) \quad [4]$$

where FFA_{FAD} is the FFA content of the FAD expressed as % MMW.

Table 6 indicates the importance of the neutral oil loss to the profitability of a coconut oil refining plant. The following formula was derived to calculate the profit-loss (PL) from the FFA content of the FAD and coconut oil:

$$\text{PL} = \frac{(\text{PR}_{\text{oil}} - \text{PR}_{\text{FAD}})(\text{FAD flow}_{\text{ACTUAL}} - \text{FAD flow}_{\text{REFERENCE}})}{100} \quad [5]$$

where PR_{oil} and PR_{FAD} are the prices of the refined oil and the FAD (U.S. \$/ton), respectively, $\text{FAD flow}_{\text{ACTUAL}}$ is the actual FAD flow (%), and $\text{FAD flow}_{\text{REFERENCE}}$ is the FAD flow of a reference (%). In comparing a reference FFA of the FAD of 80% with an actual FFA of the FAD of 70%, the $\text{FFA}_{\text{OIL IN}}$ of 3% and $\text{FFA}_{\text{OIL OUT}}$ of 0.05%, the PL is 2.4 U.S. \$/ton.

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