Production of Cocoa Butter Substitutes *via* Two-Stage Static Fractionation of Palm Kernel Oil

G. Calliauw[a,b,*], I. Foubert[a], W. De Greyt[b], P. Dijckmans[b], M. Kellens[b], and K. Dewettinck[a]

[a]Laboratory of Food Technology and Engineering, Department of Food Safety and Food Quality, Faculty of Bioscience Engineering, Ghent University, 9000 Gent, Belgium, and [b]De Smet Engineering, The Corporate Village, Zaventem, Belgium

ABSTRACT: As are traditional fractionation technologies, static dry fractionation is a highly reliable technology for the consistent production of good-quality palm kernel stearin (PKS) for use as cocoa butter substitute (CBS) after total hydrogenation. A new process route now permits the production of unhardened yet high-quality CBS. Also an increase in total stearin yield can be achieved, *via* a successful refractionation of palm kernel olein. DSC analysis together with pilot static fractionation trials on the palm kernel olein indicates that a cooling water temperature that is too low (e.g., 17°C) may result in the quick formation of unstable crystals that are possibly later converted to a more stable form. The resulting mixture of crystals with a possibly different polymorphic structure is easily squeezed through the filter cloth during filtration, whereas a slower, but more homogeneous co-crystallization occurs at higher temperature (18°C or higher) and results in a much more stress-resistant slurry. Polarized light microscopy analysis confirmed that crystal size is not the only determining factor for a successful filtration. The total two-stage static fractionation of palm kernel oil (PKO) [iodine value (IV) 18] on a pilot scale results in the following three end products: PKS IV 5 (yield: 29%, for direct use as CBS), PK olein IV 27 (yield: 58%), and PKS IV 7 (yield: 13% for use as CBS after full hydrogenation). The unhardened PKS IV 5 has outstanding melting and crystallization properties, comparable to traditional hydrogenated stearin fractions. Therefore, rather than the higher stearin yield, the reduced hydrogenation capacity is most probably the most important benefit of the two-stage static fractionation process.

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KEY WORDS: Cocoa butter substitutes, crystallization, DSC, palm kernel oil, squeeze through, static fractionation.

Owing to its high content of lauric and myristic acids, palm kernel oil (PKO) is well known as a suitable feedstock for the production of confectionery fats (1). For these applications the refined oil is commonly modified or blended. The highest-quality, steepest-melting fats resulting after modification are usually called cocoa butter substitutes (CBS) and are ideal to produce compound chocolate (2). These substitutes show physical properties similar to cocoa butter but differ considerably chemically. Hence, these fats have very little compatibility with cocoa butter (3). Besides cocoa butter-like physical behavior, high-quality CBS possess a good flavor release and show sufficient bloom resistance. Unlike cocoa butter, usually no tempering is needed during the production of confectionery (1,4).

The modification from PKO to CBS is traditionally carried out *via* a combination of fractionation and hydrogenation. The former technology is still of great interest in the oil and fats industry. The basis of a fractionation is a fractional crystallization followed by a separation between the liquid olein fraction and the crystallized stearin fraction (5–7). Basically, there are three main strategies within this technology: dry fractionation, solvent fractionation, and detergent fractionation (7). For the latter two methods, a solvent (hexane or acetone) or a detergent is used to improve the phase separation. Thanks to the use of advanced membrane filter presses, a dry fractionation can be conducted with an adequate phase separation (7,8). Dry fractionation is therefore the cheapest and simplest modification technology. In addition, the total reversibility and zero oil loss inherent to the process make it most attractive (9). Since the filterability of crystals is influenced by polymorphism, operational fractionation temperatures should be chosen that allow the more stable forms to be formed (10). In recent years, a new static dry fractionation technology (so-called Statoliser technology) was developed by De Smet Engineering for the dry fractionation of fats that form very viscous, nonpumpable slurries at the desired degree of crystallization. The Statoliser technology proved to be a successful technology for the production of good-quality palm kernel stearin (PKS) [iodine value (IV) 7] at a high yield, up to 40% (11).

This article describes a study on the two-stage dry static fractionation of PKO. The main objective of this research was to develop a process that resulted in an overall higher yield of good-quality PKS from which a (major) part could be used as CBS without additional hydrogenation. This feature would provide a major advantage over traditional CBS production processes from lauric oils.

For the optimization of the two-stage fractionation process, DSC and polarized light microscopy were used. DSC is a very useful analytical technique because it can simulate static (isothermal) dry crystallization.

MATERIALS AND METHODS

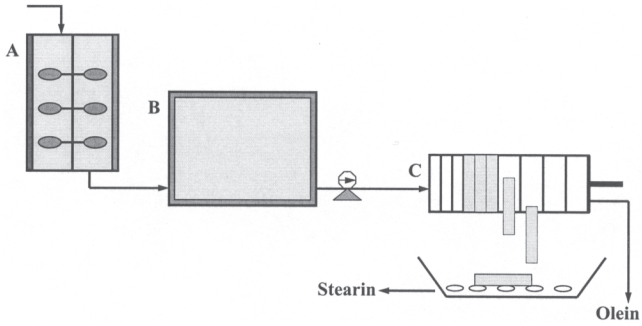
Materials. Refined, bleached, deodorized PKO (IV 18.2) was used as feedstock for the fractionation trials. To characterize the feedstock, the solid fat content (SFC) and FA profile were determined by pulsed NMR according to the AOCS Cd 16-81 non-

*To whom correspondence should be addressed.
E-mail: CAG@DesmetGroup.com

TABLE 1
FA Composition[a] and SFC Data[b] of Original RBD Palm Kernel Oil (PKO)

FA composition (%)	RBD PKO	SFC	RBD PKO
C8:0	3.1 ± 0.2	10°C	76.2 ± 0.5
C10:0	3.1 ± 0.1	15°C	57.1 ± 0.6
C12:0	47.5 ± 0.1	20°C	39.5 ± 0.7
C14:0	16.2 ± 0.2	25°C	20.5 ± 0.3
C16:0	8.6 ± 0.1	30°C	0.0 ± 0.0
C18:0	2.2 ± 0.1	35°C	0.0 ± 0.0
C18:1	16.6 ± 0.2		
C18:2	2.7 ± 0.0		

[a]%, mean ± SD of three samples. RBD, refined, bleached, deodorized.
[b]SFC, solid fat content, wt%, mean ± SD of three samples.



SCHEME 1

tempered serial method (12) and by GC with the AOCS official method Ce 1-62 (12) (further explained in the Methods section) (Table 1).

Pilot fractionation equipment and experimental design. Static fractionation trials were conducted in a pilot static fractionation unit (De Smet Engineering), which is outlined in Scheme 1. After a short crystal initiation in the dynamic crystallizer (A), the oil was transferred to the static crystallizer (B) in which the oil crystallized further under static conditions (no agitation) for a certain time at a selected temperature program. The resulting slurry was taken by a mono-screw pump to the membrane press filter (C). Liquid olein was collected continuously during the filtration process and recovered *via* an olein drain system. The solid stearin fraction was collected in a hopper after the filter was opened at the end of the filtration. In the remainder of this article, temperatures referring to fractionation conditions are cooling water temperatures and not the actual oil temperatures during fractionation. Experimental conditions applied during the single stage (reference) trial and two-stage static fractionation trials are given in Table 2.

PKS fractions were fully hydrogenated in a pilot scale Parr 4500 Pressure Reaction Apparatus with a useful capacity of 2000 mL under following process conditions: temperature, 175–200°C; H_2 pressure, 2.5–3 bar; time, 1.5–2.5 h; agitation, 60 rpm; catalyst, 0.5% (w/w) Nysofact 101 IQ (Engelhard, De Meern, The Netherlands).

Methods. The FA were esterified with methanol in the presence of potassium hydroxide as catalyst, and the methyl esters were then separated by GLC according to the AOCS official method Ce 1-62 (12). The determination of the clear m.p. (CMP) of the stearins was executed with the use of a Mettler FP80 central processor and a Mettler FP81 capillary melting point unit (Mettler-Toledo). The capillaries were filled with ±10 mm of totally liquid samples and sealed in a flame. Then they were held at −18°C for 1 h. For these stearin samples, the starting temperature of 20°C was then elevated at 1°C/min. The CMP was then determined as the first intersection of the curve with the baseline. The IV was determined using the AOCS official recommended method Cd 1b-87 (12). The DSC experiments were performed with a DSC TA Q1000 (TA Instruments). The DSC was calibrated with indium, azobenzene (Sigma-Aldrich, Bornem, Belgium), and undecane (Acros Organics, Geel, Belgium) prior to analyses. Nitrogen was used to purge the system. PKO (fraction) samples (5–15.0 mg) were sealed in hermetic aluminum pans using the following sample preparation, method B as described by Foubert *et al.* (13). An empty pan was used as a reference. For the isothermal experiments, the applied time–temperature program was: holding at 65°C for 15 min to ensure a completely liquid state, cooling at

TABLE 2
Process Parameter Data of Pilot-Scale Trials

	Single stage	Two stage										
		First stage					Two stage					
	1	2	3	5	6	7	4	8	9	10	11	12
Program	4 h 18°C	4 h 20°C	4 h 23°C + 1 h 20°C	4 h 22°C + 1 h 20°C	4 h 22°C	4 h 22°C	4 h 17°C	4 h 19°C	4 h 18°C	4 h 17°C	4 h 20°C	4 h 21°C
T slurry (°C)	22.5	24.6	24	24.8	23.9	25	20.5	21.6	21.5	21	22.7	22
SFC slurry (%)	31	21	24.5	22.2	18.6	21	21.6	18.5	19.4	22.1	17.4	18.3
SFC cake (%)[a]	75.2	78	78.9	83	75	78	64.7	78.1	76	69.5	75	67.5
$Yield_{SFC}$ (%)	41.2	26.9	31	26.7	24.8	26.9	33.4	23.7	25.5	31.8	23.2	27.2
$Yield_{weight}$ (%)	41	25.9	28.9	22	24.6	24.6	30.7	21.3	19.3	20	19.5	22.8
IV olein	25.5	23.4	23.9	23.7	23.4	23.4	27.6	27.2	26.8	27.3	27	25.9
IV stearin	7.1	6	5.2	4.8	5.9	5.2	11.9	7.4	7.9	8.9	7.1	6.2
$Yield_{IV}$ (%)	39.7	28.2	30.5	28	29.7	28.7	24.8	19.6	18	21.2	18.1	12.7

[a]Squeezing pressure, 25 bar; chamber width, 25 mm.

8°C/min to the selected isothermal temperature (±0.05°C), and holding at that temperature until crystallization had finished. For the nonisothermal experiments the following time–temperature program was used: holding at 60°C for 15 min, cooling at 5°C/min to -60°C, holding at -60°C for 5 min, heating at 5°C to 60°C. Polarized light microscopy analyses were conducted with a Leitz Diaplan Light Microscope equipped with a Linkam temperature control system and a Wild MPS camera with a Photoautomat MPS (Leica, Wetzlan, Germany).

RESULTS AND DISCUSSION

Single-stage static fractionation of PKO (reference trial–trial 1). A reference trial was conducted by holding the PKO (IV 18)—after crystal initiation—for 4 h in the static fractionation unit at 18°C cooling water temperature (isothermal crystallization). Filtration of the slurry (temperature, 22.5°C; SFC, 31%) resulted in a stearin fraction with IV 7.1 at a yield of 41%. The IV of the corresponding olein was 25.5. The PKS thus obtained is termed "Reference Stearin" in the rest of this report and is used as reference sample for the evaluation of the quality of the other stearin samples further obtained in the study.

Two-stage static fractionation of PKO: (i) First stage—production of PKS with IV 5. The objective of the first stage of the two-stage fractionation process is the production of a PKS fraction with IV ~ 5. Theoretically, this can best be achieved by applying a (slightly) higher cooling water temperature in the static crystallizer. This would result in less (and more selective) crystallization and hence in a harder stearin fraction (lower IV, but also lower yield). The residence time was kept constant (4 h), but the cooling conditions differed (different isothermal temperature or cooling curves) during the trials (Table 2). The best results were obtained during trial 7 when PKO was held for 4 h at 22°C. Filtration of the slurry (temperature, 25°C, SFC, 21%) resulted in a stearin fraction with IV 5.2 at a yield of 29%. The IV of the corresponding olein was 23.4, which is ±2 units lower than the IV of the olein obtained in the reference trial.

PKS with IV 5 is identified further in this study as "First Stage Stearin." Comparison of the quality of the different stearin samples is given later in the paper.

The palm kernel olein obtained during trials 2, 3, 6, and 7 was blended and used as feedstock for the second-stage refractionation trials.

(ii) Second stage—production of IV 7. In the initial test 4 (Table 3), the cooling water temperature in the static crystallizer was set at 17°C. This lower temperature (compared with the first-stage fractionation) was assumed to be necessary since (most of the) high(er)-melting TG are already separated during the first-stage fractionation. However, these conditions (4 h at 17°C) did not result in good crystals and, hence, crystals were squeezed through the filter cloth. The olein could not be squeezed out sufficiently, resulting in a stearin fraction with a far too high IV of 12.

Squeezing through or fat exudation is a known phenomenon (8), but at the same time it is poorly understood. It can be caused by the presence of unstable crystals, by the presence of small crystals, and/or the presence of too many small crystals. Crystal size is, however, probably not the only determining factor since this would imply that squeezing through occurs more in the case of smaller crystals. From practice, it is known that this is not always the case.

Rather than trying to solve this problem by trial-and-error, we decided to simulate the isothermal static refractionation of palm kernel olein by DSC. This technique was selected together with polarized light microscopy to study the crystallization and the stability of the different polymorphic forms in a more fundamental way.

Crystallization of PKO and palm kernel olein was first studied by polarized light microscopy. In both products, crystallization starts with the formation of small spherulitic crystals (*ca.* 10–50 μm). In a later stage, formation of bigger spherulitic crystal structures (*ca.* 200—300 μm), growing rather concentrically, is observed in both palm kernel olein held at 17°C and PKO held at 24°C (Fig. 1). The microscopic crystal structure is very similar, with some smaller crystals present in the PKO slurry. Filtration of this PKO slurry gives no problem, while significant squeezing through is observed during filtration of the palm kernel olein slurry. This observation is a confirmation that crystal size is not the only determining factor for a successful membrane press filtration.

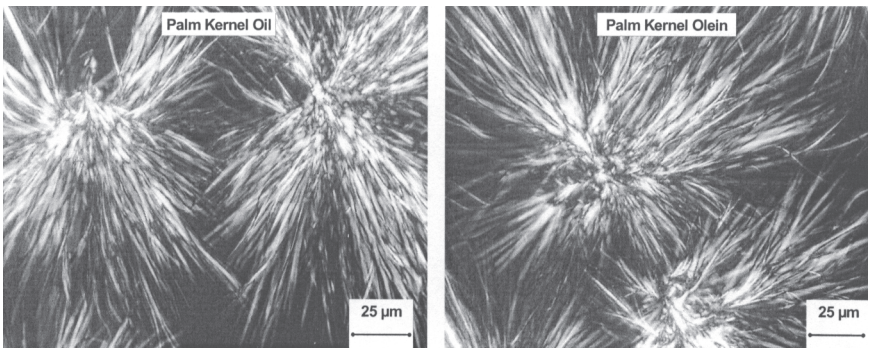


FIG. 1. Polarized light microphotograph of static crystallization of palm kernel oil at 24°C and palm kernel olein at 17°C, both after 1 h.

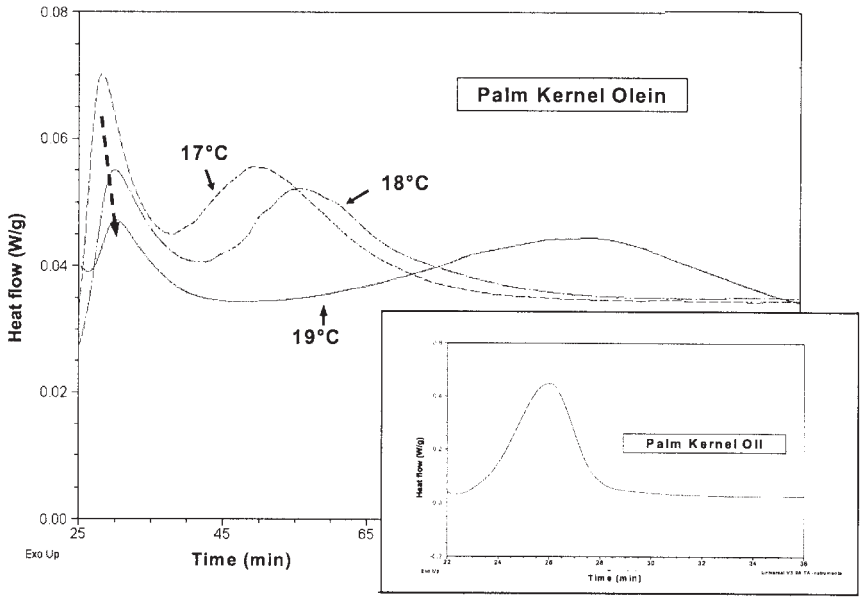


FIG. 2. Isothermal static crystallization thermograms of palm kernel olein (at 17, 18, and 19°C) and palm kernel oil (at 14°C).

Simulation of refractionation by DSC. The DSC experiments showed a clear difference in crystallization behavior between PKO and palm kernel olein. Whereas the PKO crystallizes in just one continuous step (Fig. 2), the palm kernel olein (Fig. 2) shows a two-step crystallization at lower temperature (e.g., 17°C). The first peak loses relative importance to the second peak as the isothermal temperature rises (e.g., 19°C).

This two-step crystallization at lower temperature can be due to two phenomena, either: (i) the crystallization of a less stable polymorph followed by the crystallization (from the melt or *via* polymorphic transition) of the more stable polymorph; or (ii) a fractionated crystallization where at first only the high-melting TG crystallize, followed afterward by the crystallization of the lower-melting TG.

The fact that the magnitude of the first peak gradually decreases vs. the second peak as crystallization temperature rises (from ~50% of total area to less than 20%) could be an indication for the presence of different polymorphs because, in the case of fractionated crystallization only, a relative increase of the first peak (higher-melting fraction) would be expected.

To find out whether the first crystallization peak in the DSC curve is really due to the fast crystallization of an unstable polymorph, a stop-and-return experiment was executed on the same sample. In this experiment, the oil is immediately melted after occurrence of the first crystallization peak. In this way the magnitude and the temperature(s) of the melting peak(s) of the totally crystallized sample and the sample after the first crystallization step can be compared (Fig. 3).

Figure 3 shows that the melting curve of the totally crystallized sample has only one melting peak. Moreover, both temperature ranges (of the totally crystallized sample and the sample after the first crystallization step) are very similar to one another. If the two-step crystallization in the olein is actually a fractional crystallization, the melting of the sample after the first crystallization step would be supposed to happen at higher temperatures than the melting of the totally crystallized sample does. If the occurrence of the first peak is due to the crystallization of an unstable polymorph, a melting peak at lower temperatures would be expected. Neither is the case. Consequently, this experiment does not confirm the hypothesis of a fast crystallization in an unstable polymorph, followed by a slower crystallization in a more stable form. However, this hypothesis may not be rejected either since it remains possible that the supposedly un-

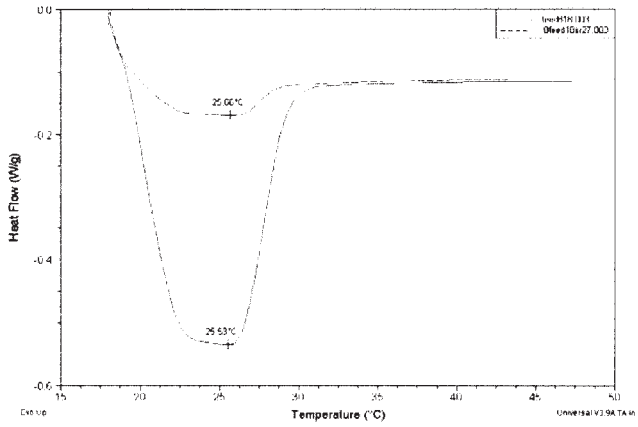


FIG. 3. Stop-and-return heating thermogram of palm kernel olein's first and total crystallization peak (after 43 and 100 min, respectively). Temperatures of melting peaks are indicated.

TABLE 3
Iodine Value, CMP, FA Composition, and SFC Data of Different Fractionated and Hydrogenated Palm Kernel Stearins[a]

	One-stage fractionation		Two-stage fractionation		
	Reference stearin		First-stage stearin	Second-stage stearin	
Parameters	Native	Hydro[b]		Native	Hydro[b]
Iodine value	7	<1	4.8	7.4	<1
CMP (°C)	33	38	35	32	39
FA composition[a]					
C10:0	2.2 ± 0.2	2.6 ± 0.1	2.2 ± 0.0	2.8 ± 0.1	3.1 ± 0.1
C12:0	54.5 ± 0.1	53.4 ± 0.4	54.9 ± 0.1	56.3 ± 0.1	55.9 ± 0.1
C14:0	23.2 ± 0.1	22.2 ± 0.1	25.6 ± 0.1	19.6 ± 0.1	19.3 ± 0.0
C16:0	9.8 ± 0.1	9.3 ± 0.1	10.1 ± 0.0	8.9 ± 0.2	8.8 ± 0.2
C18:0	2.2 ± 0.0	10.5 ± 0.1	2.0 ± 0.0	2.0 ± 0.1	10.9 ± 0.1
C18:1	6.9 ± 0.1	—	4.7 ± 0.2	7.5 ± 0.1	—
SFC[c]					
10°C	92.3 ± 0.2	99.3 ± 0.4	97.2 ± 0.2	92.6 ± 0.3	98.9 ± 0.2
20°C	86.7 ± 0.5	98.1 ± 0.4	94.8 ± 0.2	82.9 ± 0.6	96 ± 0.3
25°C	74.7 ± 0.5	92.0 ± 0.3	84.9 ± 0.3	63.0 ± 0.3	84 ± 0.3
30°C	34.1 ± 0.7	50.3 ± 0.5	56.1 ± 0.8	16.3 ± 0.7	38 ± 0.5
35°C	0.0 ± 0.0	4.8 ± 0.2	1.1 ± 0.2	0.0 ± 0.3	5.1 ± 0.3

[a]CMP, clear melting point; FA composition (wt%), mean ± SD of three samples; SFC, mean ± SD of three samples. For other abbreviation see Table 1.
[b]Reference and second-stage stearin are fully hydrogenated.
[c]Serial measurements, no thermal pretreatment (AOCS Cd 16-81) (ref. 12).

stable crystals rapidly convert to their stable polymorphic form during heating.

Thus, squeeze-through appears to be linked to the occurrence of a pronounced two-step crystallization in the oil, which is the case for palm kernel olein at lower crystallization temperatures (<17°C). Static fractionation at 18 and 19°C (Trials 8 and 9, Table 2) gives no problems of squeezing through, although a smaller first crystallization peak can still be observed in the DSC curves (Fig. 2). This means that a two-step crystallization does not necessarily lead to filtration problems as long as the first peak is not too pronounced.

DSC and polarized light microscopy analyses were not fully conclusive about the origin of the squeeze-through problem observed during the isothermal fractionation of palm kernel olein at 17°C. Nevertheless, these analyses showed that a homogeneous, one-step crystallization (co-crystallization) with formation of stable and sufficiently large crystals gives the highest chance for a good filtration. This can be obtained by keeping the slurry at a slightly higher fractionation temperature (e.g., 19–20°C).

Two-stage static fractionation of PKO: Second stage—production of IV 7 (further tests). Based on the results of the DSC analyses, additional static refractionation trials of palm kernel olein were conducted. In the different tests, the slurry was kept for 4 h at varying constant temperatures (18–21°C). Best results in terms of both stearin quality and yield were obtained at crystallization temperatures of 19 and 20°C (Trials 8 and 11, Table 2). At the latter temperature, yield was a little lower (19.5%) but the stearin obtained was very similar to the Reference Stearin. The stearin of trial 8 was named "Second Stage Stearin." The corresponding olein fraction had an IV of 27, which is 1.5 units higher than the olein obtained in the reference trial. This shows that higher-melting TG are more efficiently removed in a two-stage fractionation process compared with a one-step fractionation.

The characteristics of this Second Stage Stearin are further compared with the other PKS fractions (Reference and First Stage Stearin) in the next section.

Evaluation of the physicochemical properties of different (hydrogenated) PKS. The physicochemical characteristics of different native and fully hydrogenated PKS fractions are given in Table 3. Most interesting with respect to their (potential) use as CBS is to compare the characteristics of the fully hydrogenated reference, the second-stage stearins (IV < 1), and the unhardened first-stage stearin (IV 4.8). SFC curves indicate a high SFC at 20°C (94–98%) and a steep melting curve between 25 and 35°C for all three products. The main difference in SFC is observed at 30°C. At this temperature, the fully hydrogenated Reference Stearin and the native first-stage stearin still have a high SFC (50 and 56%, respectively). The fully hydrogenated second-stage stearin has a much lower SFC of 38%. In addition, both hydrogenated stearin samples still have ±5% SFC at 35°C, whereas the native first-stage stearin (with IV 4.8) is virtually completely melted. Hence, the CMP of the hydrogenated reference and second-stage stearin are higher than the CMP of the native first-stage stearin (38, 39, and 35°C, respectively). A CMP above 34°C has been reported as an important quality parameter for possible application as a CBS (1,14).

The crystallization and melting behavior of the fully hydrogenated reference. second-stage stearins, and the unhydrogenated first-stage stearin was also studied *via* nonisothermal

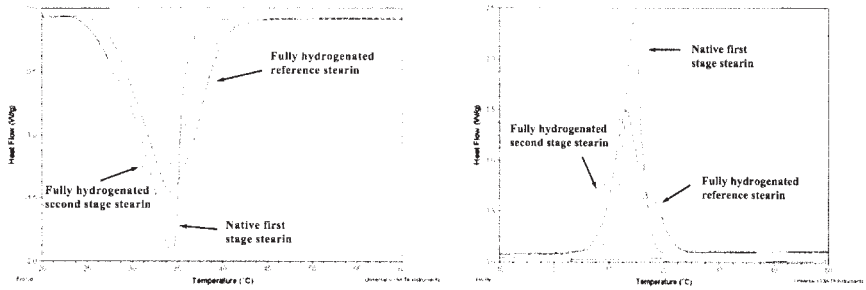


FIG. 4. Heating and cooling thermograms of palm kernel stearins.

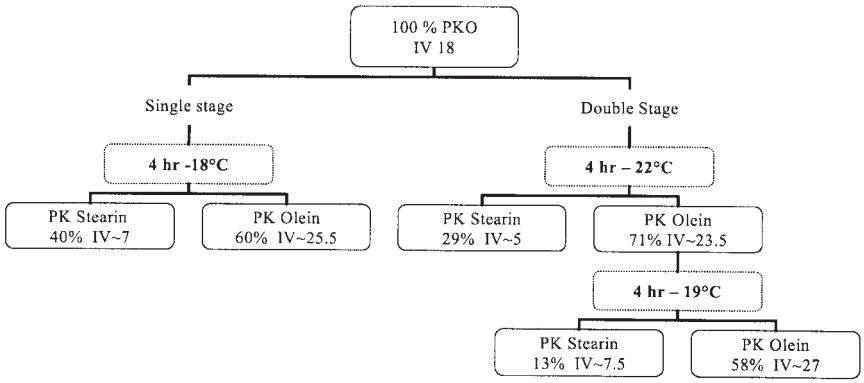
DSC. This technique was used not only to confirm the conclusions drawn from the SFC-data but also to show specific differences between the different stearin fractions. The released crystallization heat and absorbed melting heat of the hydrogenated stearins is significantly higher than those of the unhardened stearin (data not presented).

The melting curves (Fig. 4) show that for all stearins, melting starts between 26.5 and 28.3°C and that the fastest melting occurs between 32 and 35°C. The melting curve for the unhydrogenated first stage PKS (IV 4.8) is much sharper (or narrower) compared with the melting curves of the fully hydrogenated stearins. In addition, the melting curve returns more quickly and steeply to the baseline. At 36°C, the unhydrogenated first-stage PKS is completely melted, whereas the hydrogenated stearins are only fully melted at ~40°C. These observations are fully in line with the earlier reported SFC data.

Equally important for the potential use of a fat in a food formulation is its crystallization behavior. To study this, DSC can provide more useful results than SFC measurements. Crystallization curves in Figure 4 for the fully hydrogenated reference and second-stage stearin show a crystallization that starts at 21–22°C and then proceeds over a broad temperature interval. Most crystallization occurs at 12–13°C. Multiple fractions (shoulders) are apparent. On the other hand, the native first-stage stearin has a totally different crystallization curve: a sharp crystallization that starts significantly at 16.5°C with most crystallization occurring at 14.7°C.

This rapid crystallization of lauric-based fats in industrial applications has repeatedly been reported as a considerable advantage for the production of compound chocolate from CBS (15). Therefore, the sharp melting and crystallization behavior makes the first-stage PKS (IV 4.8) suitable for use as a cocoa butter substitute in specific food products.

Thus, two-stage static fractionation of PKO was optimized and compared with the traditional one-stage process in terms of yield and quality of the resulting stearin fractions. Both process routes are represented in Scheme 2. The two-stage static fractionation of PKO (IV 18) on pilot scale resulted in the following three end products: PKS IV 5 (yield: 29%, for direct use as CBS), palm kernel olein IV 27 (yield: 58%), and PKS IV 7 (yield: 13% for use as CBS after full hydrogenation). Compared with the single-stage static fractionation, the two-stage process has two potential advantages: first, there is a slightly higher stearin yield (>40%), and second, significantly less hydrogenation capacity is required: only ±30% of the PKS needs to be hydrogenated for use as CBS. Rather than the higher stearin yield, the reduced hydrogenation capacity is probably the most important benefit of the two-stage static fractionation process.



SCHEME 2

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