Zero-*trans* Shortening Using Palm Stearin and Rice Bran Oil

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ABSTRACT: Several pilot-scale trials reported in this paper, using palm stearin–rice bran oil (PS-RBO) blends, obviously did not contain *trans* FA (TFA), whereas the commercial products were found to contain 18–27% TFA. The effects of processing conditions such as rate of agitation, crystallization temperature, and composition of the blends on the crystal structure of shortenings were studied. The products were evaluated for their physicochemical characteristics using DSC, X-ray diffraction (XRD), HPLC, and and FTIR techniques. The formulation containing 50% PS and 50% RBO showed melting and cooling characteristics similar to those of hydrogenated commercial "vanaspati" samples. Analysis of the FA composition revealed that the formulated shortenings contained 15–19% $C_{18:2}$ PUFA. Tocopherol and tocotrienol contents of the experimental shortenings were in the range of 850–1000 ppm with oryzanol content up to 0.6%. XRD studies demonstrated that the crystal form in the shortenings was predominantly the most stable β′ form, and there was less of the undesirable β form.

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KEY WORDS: DSC, margarine crystallizer, palm stearin, polyunsaturated fatty acids, rice bran oil, solid fat content, *trans*-fatty acids, zero-*trans* shortening.

The influence of dietary fat on human health and nutrition is related to the quantity of fat consumed, FA composition, and the bioactive micronutrients present. Newer techniques for processing fats and oils for human consumption are needed to produce a nutritionally wholesome end product to satisfy present-day health-conscious consumers. *Trans*-FA (TFA) are now considered a risk factor for cardiovascular diseases (CVD) (1,2). Although the FA profile is a primary concern, the micronutrients present in fats and oils, such as tocopherols (soybean and corn oils), tocotrienols (palm and rice bran oils), oryzanol (rice bran oil), sesamin and sesamolin (sesame oil), and polyphenols (olive oil) modulate diseases such as CVD, cancer, diabetes, and cataracts through their ability to scavenge reactive oxygen species generated by tissue oxidation (3–5). Substantial quantities of these bioactive phytochemicals are either destroyed or removed by conventional oil processing techniques. Formulation of selected fats and oils and alternative methods of formulation therefore need to be developed to meet the nutritional and health requirements imposed on fats and oils.

Shortenings are plastic fats with solid fat crystals that hold liquid oil, thus imparting plasticity to the product. The consistency and functional properties of a fat depend on the solid–liquid ratio and crystal structure (6). The conventional method of producing shortenings employs the hydrogenation of liquid oil or blends of oils. By manipulating hydrogenation, desired properties are achieved for specific applications (7). In the Middle East, South, and Southeast Asian countries (particularly India), a shortening-like product called "vanaspati" is made by the hydrogenation of blended oils. In the Indian context, vanaspati is widely used as an inexpensive substitute for ghee (dehydrated butter oil), which has a grainy structure that is similar to vanaspati. Generally, hydrogenated fats contain 20–40% TFA that are primarily responsible for the polymorphic forms of fat crystals (α, β′, β). Of the polymorphic forms, β′ crystals are the most desirable because of their large surface area, fine arrangement (packing of small crystals), and greater oil-holding capacity; the amount of β′ crystals is a function of TFA, solid fat content (SFC), fat blends, and the crystallization process employed (8). More than 90% of the TFA in foods is produced by hydrogenation, which imparts firmness to shortening and margarines through the saturation of double bonds and conversion of other double bonds to the *trans*-configuration. In producing shortening without using hydrogenation, the selection of fat blends and crystallization process is critical. Few patents or reports are available on commercially feasible processes for producing zero-*trans* shortening. Kok *et al.* (9) prepared *trans*-free margarine from a soybean oil fraction through interesterification on a pilot scale followed by crystallization using an Armfield FT 25 B crystallizer. These authors established the feasibility of producing a zero-*trans* margarine without hydrogenation that is organoleptically and functionally similar to commercial margarine. Nor Aini *et al.* (10) and Reddy and Jeyarani (6) demonstrated a bench-scale process for *trans*-free shortening. A simple ternary blend of palm oil–palm stearin (PS)–palm olein was subjected to physical and chemical characterization using DSC and X-ray diffraction (XRD) to demonstrate the process of preparing shortening comparable to vanaspati in terms of its functional properties (10). They recommended a palm oil–PS–palm olein blend in the ratio 80:5:15 for the Malaysian market and 60:20:20 for the Middle East markets. Reddy and Jeyarani used mango kernel and mahua fat fractions and their blends without crystallization to prepare *trans*-free bakery shortenings. They estimated SFC and enthalpy changes with DSC.

In this paper our objectives were to develop a process for producing shortening without TFA and to formulate a fat blend rich in bioactive phytochemicals without sacrificing functional properties. The product should also be capable of replacing the

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vanaspati widely used as shortening in India, Southeast Asia, and the Middle East. There are two main oils commercially available in India, palm oil and rice bran oil. Palm oil and PS, because of their FA profile and glyceride structure, provide β′ crystals under proper process conditions. Rice bran oil and palm oil are rich in tocotrienols, oryzanol, and carotenes. Blends of these would, conceptually, provide functional and nutritional properties.

MATERIALS AND METHODS

Raw materials. Crude palm oil (CPO) with 3% FFA was procured from Pothepally (Andhra Pradesh, India). Palm stearin was obtained from crude palm oil by crystallization in the pilot plant of the Agro Processing Division. Refined rice bran oil was procured from the local market.

Chemicals. Standards of FAME (Sigma, St. Louis, MO), tocopherol, tocotrienol, and oryzanol (Sigma; Merck, Darmstadt, Germany) were used for identification. HPLC-grade solvents (Merck) were used for HPLC analysis. All other chemicals used were laboratory grade.

Process equipment. A scraped-surface heat exchanger (margarine crystallizer, FT 25B; Armfield, Ringwood, Hampshire, United Kingdom) was used. The unit consists of a refrigeration system, mutator, pinworker, and resting tube complete with a microprocessor-based process control console to vary feed rate, rotational speed of the mutator and pinworker, and temperature.

Processing. Based on the melting characteristics, obtained from bench-scale experiments, only selected blends were used in scaleup studies. Liquefied PS (60°C) and refined RBO were mixed in mass ratios (PS/RBO) of 50:50 (A_1), 60:40 (A_2), and 65:35 (A_3). The blends were brought to 70°C and stirred for 10 min. The refrigerant temperature of the margarine crystallizer was adjusted to 20–27°C, so that the equilibrium temperature of the product at the inlet barrel of the mutator was 50°C. The homogeneous blends at 70°C were fed at 10 kg/h to the scraped-surface heat exchanger by the diaphragm pump. The pressure inside the mutator was adjusted to 8–9 bar, enabling crystallization to the required level at 29°C. The mutator speed was adjusted at 100, 200, and 250 rpm. The material exiting the heat exchanger was then allowed to enter the pinworker. The pinworker speed was 50, 100, and 150 rpm. The material exiting the pinworker was passed through a resting tube, and the semisolid shortening was filled at different crystallization temperatures in 500-mL closed plastic containers and tempered at 30 ± 1°C for 5 d. A summary of the trials is presented in Table 1. For comparison, two commercial samples of leading brands of vanaspati marketed in India were used as controls (C_1 and C_2).

DSC. Melting and crystallizing characteristics of the shortenings were studied using a DSC (DSC 821; Mettler Toledo, Schwerzenbach, Switzerland). Samples were subjected to the following temperature program: holding at 80°C for 30 min, cooling from 80 to –30°C at a rate of 10°C/min, and holding at –30°C for 2 min. The same sample was then heated from –30 to 80°C at the rate of 5°C/min (11). Heating and cooling thermograms were recorded. For SFC calculation, the heating thermogram at the temperature range of –30 to 60°C was used. The percentages of liquid at various temperatures were obtained directly from the DSC-821 software library. The SFC was calculated from the percentage of liquid, and melting profiles were prepared by plotting SFC against temperature (6,12).

XRD. The polymorphs of fat crystals were determined by XRD using a Philips 1710 X-ray diffractometer (Almelo, The Netherlands) emitting Cu-Kα radiation (λ = 1.5418 Å). Data were collected from 5 to 45 2θ° with a step width of 0.04° and step time of 1 s operating at room temperature. X-ray data were processed by a computer programmed to calculate absorption intensity–background, intensity, and peak width in degrees for each crystalline form and the relative contents of β and β′ crystals. The β form was calculated from the intensity of the short spacing at 4.6 Å. The β′ polymorph was calculated from the intensities of the short spacing of 3.8 and 4.2 Å. The α form was calculated from the intensity of the short spacing of 4.15 Å.

FA composition. FAME were prepared by esterifying with alcoholic sulfuric acid reagent according to the IUPAC procedure (13). A Hewlett-Packard 5890 series 11 GC (Avondale, PA) equipped with an FID was used for GC analysis of the methyl esters. Methyl esters were analyzed on a Hewlett-Packard free fatty acid phase (cross-linked FFAP) column (30 m × 0.53 mm × 1.0 μm). The injection and detector temperatures

TABLE 1
Formulation and Process Variables Employed at Pilot-Scale Processing of Shortening

Trials	PS/RBO[a] (w/w)	Refrigerant temperature (°C)	Mutator speed (rpm)	Pinworker speed (rpm)	Filling temperature (°C)
1	50:50	27	250, 200, 100	150, 100, 50	30.5
2	50:50	25	250, 200, 100	150, 100, 50	29.8
3	50:50	22	250, 200, 100	150, 100, 50	28.7
4	60:40	27	250, 200, 100	150, 100, 50	31.2
5	60:40	25	250, 200, 100	150, 100, 50	30.8
6	60:40	22	250, 200, 100	150, 100, 50	29.7
7	65:35	27	250, 200, 100	150, 100, 50	32.7
8	65:35	25	250, 200, 100	150, 100, 50	31.7
9	65:35	22	250, 200, 100	150, 100, 50	30.2

[a]PS, palm stearin; RBO, rice bran oil.

were maintained at 250 and 300°C, respectively. The flow rate of the carrier gas (nitrogen) was 20 mL/min. The oven temperature was programmed from 100 to 180°C at the rate of 5°C/min. FAME were identified by using authentic standards, and the peaks were quantified by using digital integration. FA levels were reported as relative proportions of the total composition.

Tocopherol, tocotrienol, and oryzanol contents. The analysis was performed with a Shimadzu high-performance liquid chromatograph (Kyoto, Japan) with an LC-10 AD model pump, a 7125 model Rheodyne injector (Cotati, CA) fitted with a 20 μL sample loop, an SPD-10 A UV-vis detector, with a C-R7Ae plus integrator for data acquisition and display (Shimadzu). For the analyses of tocopherols and tocotrienols, a CLC-NH2 (M) column (4.6 mm i.d. × 25 cm) (CLA, Kyoto, Japan) was used in the normal phase with the solvent system *n*-hexane/isopropanol (96:4, vol/vol) at a flow rate of 1 mL/min. The UV detector was set at 297 nm (14). Estimation of oryzanol was carried out using a μBondapak^{TM} C_{18} column (4.6 mm × 25 cm) (Waters, Milford, MA) in the reversed phase with a solvent system of acetonitrile, dichloromethane, and acetic acid (88:6:6, by vol): methanol, *n*-butyl alcohol, water (90:2:8 by vol) in the ratio of 75:25 (by vol) at a flow rate of 1 mL/min. The UV detector was set at 325 nm (15).

FTIR spectroscopy. A Nicolet Magna 560 FTIR spectrophotometer (Madison, WI) equipped with a deuterated triglycine sulfate detector that operated under Omnic version 4.1 (Madison, WI) was used for recording the infrared (IR) spectra and estimating TFA. The region of 4000 to 400 cm^{-I} was scanned with a resolution of 4.00 cm^{-1}.

Light microscopy. Crystals of the shortenings were observed with a polarized-light microscope (Nikon Optiphot) equipped with a camera (model FX-35; Nikon, Tokyo, Japan). The objective magnification was 10 × 5. A small quantity of the shortening sample (3–4 mg) was placed on a glass slide, and after placing a cover slip over it, images were taken at room temperature (30 ± 1°C).

Physicochemical characteristics. Iodine values (IV) were determined using the Wijs method according to AOCS procedure Cd 1-25, and the slip melting point (SMP) was determined in accordance with method Cc 3-25 (16).

RESULTS AND DISCUSSION

SFC. Three formulations of PS/RBO were selected based on bench-scale data. The other variables of the process, such as refrigerant temperature, mutator and pinworker speeds, and filling temperature, were selected based on their influence on the physical characteristics of the end product. More than 200 pilot-scale trials were conducted to fix the optimal process conditions.

The SFC of the experimental and commercial samples at different temperatures calculated from the DSC data are presented in Figure 1. SFC decreased sharply with an increase in temperature for all the samples, with PS/RBO 50:50 (A_1) exhibiting a SFC between commercial samples C_1 and C_2. With

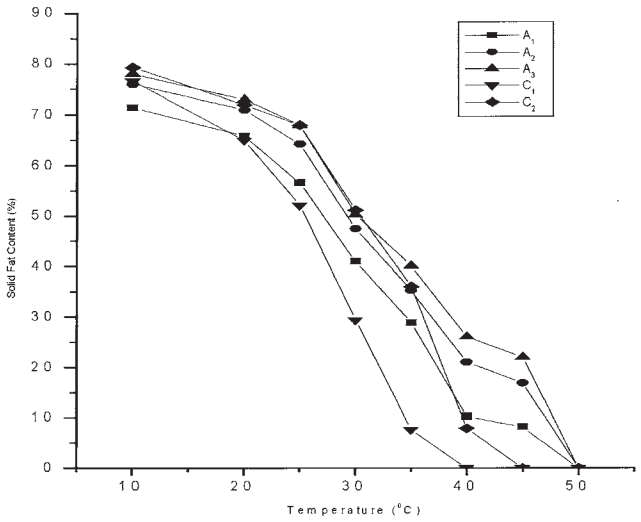

FIG. 1. Solid fat content of experimental shortenings and commercial samples. Abbreviations: A_1, palm stearin/rice bran oil (PS/RBO) 50:50 (w/w); A_2, PS/RBO 60:40; A_3, PS/RBO 65:35; C_1, commercial sample 1; C_2, commercial sample 2.

increasing PS content, the SFC increased for a given temperature. At a temperature above 35°C, the difference in SFC between the samples tended to widen. SFC profiles of shortenings affect their functional properties, and fat blends basically determine the SFC, which in turn controls the crystal structure and polymorphism under given process conditions. A steep SFC profile is indicative of a narrow plastic range, and products of this type are high-stability shortenings. This shortening therefore is not workable over a wide range of temperatures since it tends to be hard and brittle below 20°C and soft above 30°C. Shortening with 50:50 PS/RBO was comparable with the vanaspati used for deep frying and in bakery and confectionary products in India and the Middle East.

Polymorphism and crystal structure. The structures of fat crystals and their polymorphic forms were determined by using DSC, XRD, and light microscopy (Table 2, Figs. 2–6). Structure, composition, and polymorphic forms of fat crystals are the most important criteria for the functional properties of shortenings. X-ray diffraction data (Table 2) indicate that the content of α and β′ crystals tended to decrease with an increase in PS, whereas the value for the β form was higher with a higher proportion of PS. Tempering at 30 ± 1°C favored β′ crystal formation for sample A_1 and imparted a smooth consistency on storage for 5 d. The β form, although the most stable, was not desirable at higher levels because of the large crystal size, which resulted in a coarse and grainy texture. One important function of shortening is to incorporate and hold air to increase the volume of the cream or the baked cake, and β′ is mostly responsible for this property (6). The composition of A_1 had the combination of fat crystals desirable for high-stability shortening.

FA composition. Table 3 shows the FA composition of the blends and commercial products. The desirable blend, considering other physical properties, was A_1 with 50:50 PS/RBO,

TABLE 2
Polymorphic Forms[a] of Experimental Shortenings and Commercial Samples

Sample		Polymorphic forms (%)		
Code	PS/RBO	α	β′	β
A_1	50:50	27.3 ± 0.37	61.0 ± 0.68	11.7 ± 0.07
A_2	60:40	25.9 ± 0.18	59.0 ± 0.35	15.1 ± 0.28
A_3	65:35	22.8 ± 0.2	56.5 ± 0.3	20.7 ± 0.15
C_1	Comm. 1	28.5 ± 0.15	46.5 ± 0.25	25.0 ± 0.2
C_2	Comm. 2	26.0 ± 0.15	58.1 ± 0.22	15.9 ± 0.15

[a]Determined by X-ray diffraction: β, strong short spacing of 4.6 Å; β′, strong short spacing of 3.8 and 4.2 Å; α, strong short spacing of 4.15 Å. Abbreviations: C_1, commercial sample 1; C_2, commercial sample 2; for other abbreviations see Table 1. Experimental samples were crystallized at 29.8°C at a 200 rpm mutator speed and 50 rpm pinworker speed. Data are expressed as means for n = 5 samples taken from different trials.

which had a fairly balanced FA composition. This blend contained 41% $C_{16:0}$, 34.5% $C_{18:1}$, and 18.7% $C_{18:2}$. The leading commercial brands marketed in India (C_1 and C_2) had higher levels of saturated fat and lower levels of monounsaturated FA and PUFA compared with those of the experimental treatments. Predictably, sample A_1 contained no TFA, whereas the commercial samples had 18.7% and 26.7% TFA for C_1 and C_2, respectively. FTIR spectra of A_1, C_1, and C_2 samples demonstrated the extent of TFA in the commercial samples and their absence in the experimental products. The IV of sample A_1 was also significantly higher (72) than that of C_1 (69) and C_2 (49). SMP of A_1 (37°C), C_1, and C_2 were within the legal limit of 41°C prescribed for shortening and vanaspati (17).

Micronutrients. Experimental blends and commercial samples were analyzed for their tocopherols, tocotrienols, and oryzanol levels by HPLC (Table 4). Apart from their melting characteristics, the fat blends were selected to provide maximal nutritional properties in terms of micronutrients. Whereas RBO is known for its very high levels of γ-tocotrienol and γ-oryzanol, PS is also rich in γ-tocotrienol, and has a desirable melting profile. Consequently, sample A_1 had the highest level of γ-tocotrienol (631 ppm), total tocochromanols (988 ppm), and γ-oryzanol (0.6%). The fact that commercial sample C_1 had a higher level of γ-tocotrienol and a trace amount of oryzanol indicated that the tocotrienol may be mostly derived from palm oil as no other edible oil contains such a large amount of tocotrienols. The other commercial sample, C_2, was poor in both tocotrienols and oryzanol.

Physical appearance. The large number of trials conducted using the scraped-surface heat exchanger (margarine maker) were aimed at obtaining a commercially acceptable physical consistency and product stability on storage. Sample A_1 collected at 29 ± 0.8°C, was soft and homogeneous, without any liquid phase separation when tempered at 30 ± 1°C and subsequent storage at ambient conditions (25 to 30°C). Sample A_1 collected above 30°C showed liquid phase separation on storage. Although phase separation was not observed when PS content was increased (A_2 and A_3), the product became hard on storage.

Light microscopy studies were conducted to understand the physical appearance and packing of crystals as influenced by the process variables, such as composition of blend, filling temperature, and mutator and pinworker speeds. Photomicrographs (Figs. 4–6) demonstrate the results obtained therefrom. Figure 4 shows the images for 50:50, 60:40, and 65:35 PS/RBO blended shortenings crystallized at 29.8°C with the pinworker speed at 50 rpm. The 50:50 blend had larger crystals because 29.8°C was a relatively high temperature for this sample. The driving force for crystallization appeared to be

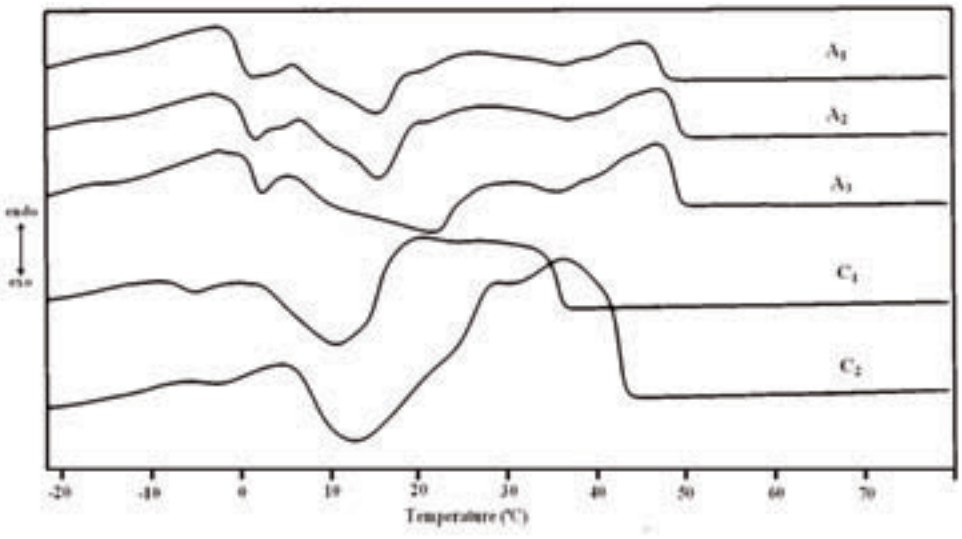

FIG. 2. DSC melting thermograms of experimental shortenings and commercial samples: A_1, PS/RBO 50:50 (w/w); A_2, PS/RBO 60:40; A_3, PS/RBO 65:35; C_1, commercial sample 1; C_2, commercial sample 2. For abbreviations see Figure 1.

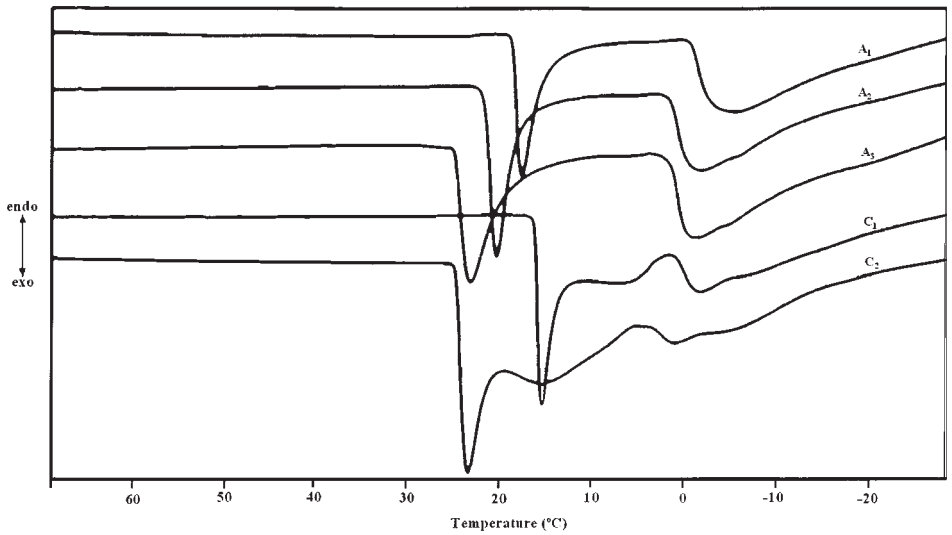

FIG. 3. DSC cooling thermograms of experimental shortenings and commercial samples: A_1, PS/RBO 50:50 (w/w); A_2, PS/RBO 60:40; A_3, PS/RBO 65:35; C_1, commercial sample 1; C_2, commercial sample 2. For abbreviations see Figure 1.

lower in this blend compared with the 60:40 and 65:35 blends. The crystal size decreased when the percentage of PS increased. Since PS is composed of high-melting TG, the appearance of more small crystals at 29.8°C could be attributed to supercooling in the 65:35 blend, which resulted in a hard consistency. The effect of crystallization temperature (30.5, 29.8, and 28.7°C) on crystal structure for the 50:50 PS/RBO blended shortening with a pinworker speed of 50 rpm is shown in Figure 5. At high temperature, fewer large crystals were formed. A lower temperature (28.7°C) favored faster

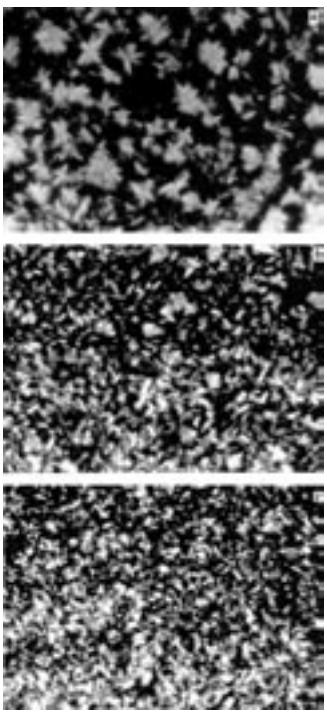

FIG. 4. Photomicrographs of (a) 50:50, (b) 60:40, and (c) 65:35 (wt%/wt%) PS/RBO blended shortenings crystallized at 29.8°C at an agitation rate of 50 rpm. For abbreviations see Figure 1.

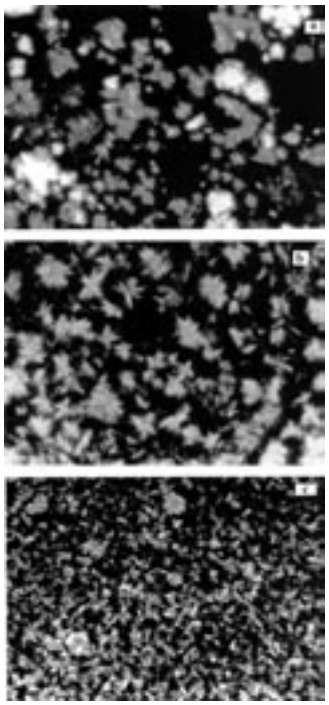

FIG. 5. Photomicrographs of 50:50 PS/RBO blended shortenings crystallized at (a) 30.5°C, (b) 29.8°C, and (c) 28.7°C.

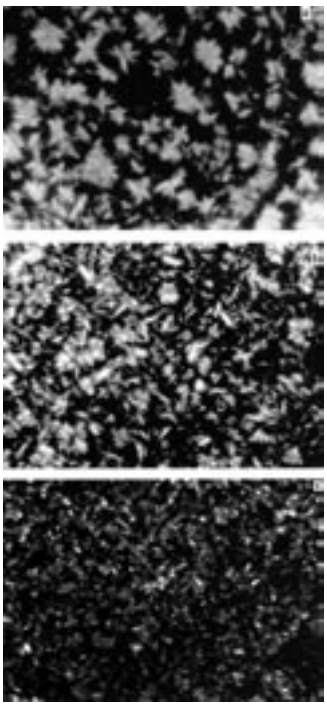

FIG. 6. Photomicrographs of 50:50 PS/RBO blended shortenings crystallized at 29.8°C, and agitation rates of (a) 50 rpm, (b) 100 rpm, and (c) 150 rpm.

nucleation, leading to smaller and more closely packed dense crystals than the product of the same blend crystallized at 30.5°C. Figure 6 shows the effect of agitation rates (50, 100, 150 rpm) on a 50:50 blend crystallized at 29.8°C. Pinworker speeds of 100 and 150 rpm resulted in a marked decrease in crystal size. When the agitation rate was low, large crystals were formed. Higher agitation rates resulted in small crystals, obviously due to the disintegration of crystals.

The SFC and fat crystals in a shortening are responsible for many of its characteristics including general appearance, organoleptic properties, oil exudation, and functional properties. The zero-*trans* shortening reported here falls in the category of a high-stability shortening with a steep SFC profile, indicating a narrow plastic range. An SFC of 10–50% in the temperature range of 40–10°C is considered desirable for a high-stability shortening (18). The A_1 blend reported here had a similar SFC profile, with 61% β′ crystals. With the increase in PS content (A_2 and A_3), the blends showed brittleness at lower temperatures with little difference in the β′ crystals. Earlier studies at bench scale using PS with various liquid oils also reported similar observations (10). The pilot-scale process developed here using the process variables mentioned elsewhere indicated that the 50:50 PS/RBO blend was ideal for producing PUFA-rich zero-*trans* shortening, and the process conditions optimized for this blend at pilot scale were 10 kg/h feed rate, 8–9 bar back pressure, 25°C refrigerant temperature, 200 rpm mutator speed, 50 rpm pinworker speed, and 29.8°C crystallization temperature.

TABLE 3
FA Composition and Chemical Characteristics of Raw Materials and Different Blends[a]

Characteristics	PS	RBO	A_1	A_2	A_3	C_1	C_2
FA (wt%)							
$C_{12:0}$	1.2	0.9	1.1	1.0	1.1	1.5	0.2
$C_{14:0}$	1.4	0.8	1.1	1.2	1.2	1.3	0.9
$C_{16:0}$	56.6	25.5	41.1	44.2	45.7	44.5	48.1
$C_{18:0}$	3.9	1.6	2.8	3.0	3.2	3.4	3.9
$C_{18:1}$	29.7	39.2	34.4	33.5	33.1	40.7	39.2
$C_{18:2}$	6.8	30.6	18.7	16.4	15.2	8.6	7.7
Others	0.4	1.4	0.8	0.7	0.5	—	—
TFA (%)	—	—	—	—	—	18.7	26.7
SMP (°C)	50.0	—	37.0	38.5	39.0	35.0	38.5
IV	42.5 ± 0.3	99.0 ± 0.7	72.0 ± 0.3	65.0 ± 0.3	63.6 ± 0.4	65.0 ± 0.5	49.0 ± 0.2

[a]TFA, *trans*-FA; SMP, slip melting point; IV, iodine value; for other abbreviations see Table 1 and 2. Values are means of three analyses.

TABLE 4
Tocopherol, Tocotrienol, and Oryzanol Contents of Experimental Shortenings and Commercial Samples[a]

Sample	α-T (ppm)	α-T_3 (ppm)	β-T_3 + γT (ppm)	γ-T_3 (ppm)	δ-T_3 (ppm)	Total T + T_3 (ppm)	Oryzanol (%)
RBO	131	297	—	933	9	1370	1.2
PS	67	126	—	314	75	582	—
A_1	104	211	—	631	42	988	0.6
A_2	93	194	—	562	48	897	0.5
A_3	90	186	—	531	51	858	0.4
C_1	185	416	—	309	39	949	0.08
C_2	58	125	18	28	9	238	0.02

[a]T, tocopherol; T_3, tocotrienol; for other abbreviations see Tables 1 and 2. Values are means of five analyses.

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