

## Chemical Interesterification of Blends of Palm Stearin, Coconut Oil, and Canola Oil: Physicochemical Properties

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**ABSTRACT:** *trans*-Free interesterified fat was produced for possible usage as a margarine. Palm stearin, coconut oil, and canola oil were used as substrates for chemical interesterification. The main aim of the present study was to evaluate the physicochemical properties of blends of palm stearin, coconut oil, and canola oil submitted to chemical interesterification using sodium methoxide as the catalyst. The original and interesterified blends were examined for fatty acid composition, softening and melting points, solid fat content, and consistency. Chemical interesterification reduced softening and melting points, consistency, and solid fat content. The interesterified fats showed desirable physicochemical properties for possible use as a margarine. Therefore, our result suggested that the interesterified fat without *trans*-fatty acids could be used as an alternative to partially hydrogenated fat.

**KEYWORDS:** *eutectic behavior, validation, multiple regressions, consistency, solid fat content, melting point*

### ■ INTRODUCTION

The challenge faced by food industries to replace *trans*-fat in numerous products lies in the development of formulations and processes that have equivalent functionality and economic viability.<sup>1</sup> To decrease or eliminate *trans*-fat in food products, alternative technological approaches such as interesterification, fractionation, and blending have been developed to replace the conventional hydrogenation process.

To achieve similar functionality in fat products like hydrogenated fat, a saturated hard stock such as palm stearin rich in palmitic acid can be used. This is added to improve tolerance to high temperatures and stability.<sup>2</sup> The production of edible fats, however, requires fat blends that are able to impart plasticity and body to the end product. This necessitates the enrichment of polyunsaturated oils, such as canola oil, into palm stearin, which basically lacks the ability to impart the required plasticity to the end product and must therefore be modified.

More recently, the concept of balancing fatty acids in oils and fats to promote health has been advocated by nutritionists and medical practitioners.<sup>3</sup> Coconut oil can improve the nutritional aspect of oils and fats due to its relatively high medium-chain fatty acids triacylglycerols content. Coconut oil is rich in saturated fatty acids (SFAs) such as lauric acid. Because of the high medium-chain fatty acids triacylglycerols content, coconut oil is a major component of infant formulas and medical foods for people who cannot absorb longer chain fatty acids.<sup>4–6</sup>

The effect of blending and interesterification (chemical or enzymatic) on the physicochemical characteristics of blends of hard fat with various oils and fats has been reported.<sup>5,7,8</sup> However, reports describing the effect of blending and chemical interesterification on the physicochemical characteristics of ternary blends are scarce.<sup>9,10</sup>

The purpose of this study was to evaluate the chemical interesterification of palm stearin, coconut oil, canola oil, and

their blends, with a view to study fat bases for application in food products. Preparation of shortenings through chemical interesterification of palm stearin, coconut oil, and canola oil is reported in the present investigation. The functionality of the finished product was based on the softening (SP) and melting points (MPs), consistency, and solid fat content (SFC) of the blends and interesterified fats. The interaction of these oils and fats and their compatibility are also discussed.

### ■ MATERIALS AND METHODS

**Materials.** Palm stearin was obtained from Agropalma S/A (Pará, Brazil), coconut oil was from Copra Alimentos Ltd.a. (Alagoas, Brazil), and canola oil was from Bunge Alimentos S.A. (São Paulo, Brazil). The fats were stored at 0 °C prior to use. All chemicals used were of either analytical or chromatographical grades.

**Blend Preparation.** Fat blends, formulated with palm stearin, coconut oil, and canola oil were mixed at different ratios, according to Table 1. Three blends represented the original components, three were binary blends, and four were ternary blends. The blends were prepared after complete melting of the fats at 70 °C and stored under refrigeration. Blends 8, 9, and 10 were used to validate the statistical model.

**Chemical Interesterification.** Chemical interesterification was performed according to ref 11 with modifications. Two hundred grams of each blend was melted in a glass jar at 85 °C under reduced pressure to limit moisture and air. The chemical reaction was started by the addition of 0.3% (w/w) sodium methoxide (Merck Co.) as the catalyst. The blends were interesterified under reduced pressure for 60 min at 88 ± 2 °C. The start of the reaction was associated with the appearance of a reddish-brown color. To terminate the reaction, 5 mL of distilled water was added. The presence of water inactivates the catalyst by converting it to methanol. Kieselhur and anhydrous

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Table 1. Compositional Design of the Blends (w/w)

blends	%		
	palm stearin	coconut oil	canola oil
1	100.0	0.0	0.0
2	0.0	100.0	0.0
3	0.0	0.0	100.0
4	50.0	50.0	0.0
5	50.0	0.0	50.0
6	0.0	50.0	50.0
7	33.3	33.3	33.3
8	66.7	16.7	16.7
9	16.7	66.7	16.7
10	16.7	16.7	66.7

sodium sulfate were added to minimize the darkening caused by the presence of a diacylglycerol metal derivative (active catalyst) and to remove residual water, respectively. The reagents were removed by filtering the samples with filter paper. The fat was poured into a glass jar and stored at 5 °C prior to use. Noninteresterified oil is abbreviated to NIE, and chemical interesterified blends are abbreviated to CIE.

**Fatty Acid Composition.** The fatty acid composition was determined after conversion of fatty acids into their corresponding methyl esters (FAMES) using the method described by the ISO.<sup>12</sup> Analyses of FAMES were carried out in a Varian GC gas chromatograph (model 430 GC, Varian Chromatograph Systems, Walnut Creek, CA), equipped with a CP 8412 autoinjector. The Galaxie software was used for quantification and identification of peaks. Injections were performed into a 100 m fused silica capillary column (i.d. = 0.25 mm) coated with 0.2 μm of polyethylene glycol (SP-2560, Supelco, United States) using helium as the carrier gas at an isobaric pressure of 37 psi; linear velocity of 20 cm/s; makeup gas, helium at 29 mL/min at a split ratio of 1:50; and volume injected, 1.0 μL. The injector temperature was set at 250 °C, and the detector temperature was set at 280 °C. The oven temperature was initially held at 140 °C for 5 min, then stepped to 240 °C at a rate of 4 °C/min, and held isothermally for 30 min. Pure oils and their blends were analyzed in triplicate, and reported values represent the average of the three runs.

Medium-chain saturated fatty acids (MCSFAs) are expressed as the sum of the amounts of caprylic, capric, and lauric acids. Long-chain saturated fatty acids (LCSFAs) are expressed as the sum of the amounts of myristic, palmitic, and stearic acids. SFAs are expressed as the sum of the amounts of caprylic, capric, lauric, myristic, palmitic, and stearic acids. Unsaturated acids (USFAs) are expressed as the sum of the amounts of oleic, linoleic, and linolenic acids. Monounsaturated fatty acids (MUFA) are expressed as amounts of oleic acid. Polyunsaturated fatty acids (PUFAs) are expressed as the sum of the amounts of linoleic and linolenic acids.

**Iodine Value (IV).** The IV was calculated from the fatty acid composition, according to the procedure described in the AOCS official method Cd 1c-85.<sup>13</sup>

**Atherogenic Index (AI).** The AI was calculated according to Kim, Lumor, and Akoh,<sup>9</sup> by the following equation:

$$AI = [C12: 0 (w/w, \%) + 4 \times C14: 0 (w/w, \%) + C16: 0 (w/w, \%)] / USFA (w/w, \%)$$

**SP.** The SP was determined by the open tube MP method, according to the AOCS official method Cc 3-25.<sup>13</sup> Three replicates of this analysis were performed.

**MP.** The MP was determined by the closed tube MP method, according to the AOCS official method Cc 1-25.<sup>13</sup> Three replicates of this analysis were performed.

**Consistency.** The consistency was determined via the penetration test using a 45° acrylic cone fitted to a constant speed model TA-XT2 Texture Analyzer, Stable Micro Systems, United Kingdom. Fat samples were heated to 70 °C in a microwave oven for complete

melting of the crystals and stored in 50 mL glass beakers (Pyrex, United States). Tempering was allowed to occur for 24 h in a standard refrigerator (5–8 °C) and then for 24 h in an oven with controlled temperature (5, 10, 15, 20, 25, 30, 35, 40, and 45 ± 0.5 °C). The tests were conducted under the following conditions: determination of force in compression; distance, 10.0 mm; speed, 2.0 mm/s; and time, 5s.<sup>14</sup> Measurements were performed in duplicate, and the reported values are the simple average of the two values. The consistency was calculated as a “yield value” (kgf/cm<sup>2</sup>), according to the equation proposed by Haighton<sup>15</sup>

$$C = \frac{K \times W}{p^{1.6}} \quad (1)$$

where  $C$  is the yield value (kgf/cm<sup>2</sup>),  $K$  is a constant depending on the cone angle (4700-undimensional),  $W$  is the compression force (kgf), and  $p$  is the penetration depth (mm/10).

**SFC.** The SFC was determined with a DSC 4000 differential scanning calorimeter (Perkin-Elmer Corp., Norwalk, CT). The data processing system used was the Pyris Series Thermal Analysis System software. An empty aluminum pan was used as a reference, and each sample was accurately weighed (5–10 ± 0.1 mg) for DSC analysis. The sample was heated to 80 °C and held for 10 min. Thereafter, the temperature was decreased at 10 °C/min to –60 °C. After it was held for 10 min at –60 °C, the melting curve was obtained by heating to 80 at 5 °C/min. The temperature and heat of fusion were calibrated with indium (onset temperature, 156.6 °C).

The SFC was obtained from the melting curve. The SFC as a function of temperature was calculated from the partial areas at different temperatures (–25 to 60 °C, intervals of 5 °C).<sup>5</sup>

**Statistical Analysis.** Results were expressed as mean ± standard deviation (SD). Differences between the samples (fatty acid composition, SP, and MP) during the experimental period were statistically analyzed using repeated measures analysis of variance (ANOVA), followed by a posthoc Tukey test, taking on  $P < 0.05$ .

Experimental results for SP and MPs, consistency, and SFC were applied to obtain the regression models, as a function of the proportions of each ingredient ( $x_1$  = palm stearin,  $x_2$  = coconut oil, and  $x_3$  = canola oil) present in the blends 1 to 7:  $\hat{y}_i = \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3$ , where  $\hat{y}_i$  = estimated response,  $\beta_i$  = coefficients estimated by the least-squares method, and  $x_i$  = dependent variables.

The quality of the models was evaluated by ANOVA and adjusted coefficient of determination ( $r^2$ ), with the optimization obtained by the Barros Netto, Scarminio, and Bruns.<sup>16</sup> Statistical analysis was performed using STATISTICA 9.0 software.<sup>17</sup> Validation was performed based on three points (blends 8, 9, and 10) in conditions of interest within the surface and applying the same experimental procedures used to construct the models.

## RESULTS AND DISCUSSION

**Fatty Acid Composition.** The fatty acid composition of the pure oil, their blends, and interesterified fats are shown in Table 2. All blends were *trans*-free fatty acids. The total saturated fatty acid of the blends with higher content of palm stearin and coconut oil was greater than 35.6%, a value that renders them highly resistant to oxidative rancidity.<sup>18</sup>

Blends prepared using palm stearin have higher concentrations of palmitic acid, which imparts a desirable smooth consistency required for application such as margarines and shortenings. The  $\beta'$ -crystal form is more stable in shortenings with higher palmitic acid contents.<sup>19</sup> Blends with a higher content of coconut oil contain predominantly lauric acid. According to Zhang, Smith, and Adler-Nissen,<sup>20</sup> fats that have a high content of lauric and myristic acids exhibit very sharp MPs. The sharp melt, low MP, and low unsaturated fatty acids content make coconut oil particularly suited as fats for low-moisture food products for applications such as confectionery

Table 2. Fatty Acid Composition (g/100 g) of Palm Stearin, Coconut Oil, Canola Oil, and Their Blends<sup>a</sup>

fatty acids <sup>b</sup> (g/100 g)	1	2	3	4	5	6	7	8	9	10
8:0	ND a	4.9 ± 0.0 g	ND a	2.2 ± 0.0 d	ND a	2.4 ± 0.0 e	1.5 ± 0.0 c	0.7 ± 0.0 b	3.1 ± 0.0 f	0.7 ± 0.0 b
10:0	ND a	3.9 ± 0.0 f	ND a	1.8 ± 0.0 c	ND a	1.9 ± 0.0 c	1.2 ± 0.0 d	0.6 ± 0.0 b	2.5 ± 0.0 e	0.5 ± 0.0 b
12:0	ND a	38.7 ± 0.0 g	ND a	18.5 ± 0.1 d	ND a	19.0 ± 0.1 e	12.4 ± 0.0 c	6.1 ± 0.0 b	25.4 ± 0.1 f	6.0 ± 0.0 b
14:0	1.1 ± 0.0 c	19.8 ± 0.0 j	ND a	10.1 ± 0.0 h	0.5 ± 0.0 b	9.7 ± 0.0 g	6.8 ± 0.2 f	3.9 ± 0.0 e	13.2 ± 0.0 i	3.3 ± 0.0 d
16:0	57.3 ± 0.0 j	12.7 ± 0.0 c	4.9 ± 0.0 a	36.4 ± 0.1 h	32.1 ± 0.1 g	8.7 ± 0.0 b	25.9 ± 0.1 f	42.7 ± 0.2 i	19.2 ± 0.0 e	15.2 ± 0.0 d
18:0	5.2 ± 0.0 j	2.2 ± 0.0 a	2.5 ± 0.0 c	3.8 ± 0.0 g	3.9 ± 0.0 h	2.3 ± 0.0 b	3.3 ± 0.0 f	4.3 ± 0.0 i	2.7 ± 0.0 d	2.9 ± 0.0 e
18:1	30.8 ± 0.0 d	12.5 ± 0.0 a	63.6 ± 0.0 j	22.1 ± 0.1 b	48.1 ± 0.1 h	38.5 ± 0.1 g	36.4 ± 0.3 f	33.7 ± 0.2 e	24.6 ± 0.0 c	50.3 ± 0.0 i
18:2	5.4 ± 0.0 a	5.5 ± 0.0 a	21.2 ± 0.0 g	5.2 ± 0.2 a	11.5 ± 0.2 d	13.5 ± 0.0 e	10.0 ± 0.7 c	7.5 ± 0.4 b	8.2 ± 0.0 b	16.1 ± 0.0 f
18:3	ND a	ND a	7.9 ± 0.0 g	ND a	3.8 ± 0.0 d	3.9 ± 0.0 e	2.6 ± 0.1 c	1.2 ± 0.0 b	1.2 ± 0.0 b	5.2 ± 0.0 f
IV <sup>c</sup>	26.5 ± 0.0 c	10.7 ± 0.0 a	112.0 ± 0.1 j	19.0 ± 0.1 b	71.3 ± 0.3 h	66.9 ± 0.1 g	55.4 ± 0.8 f	45.1 ± 0.5 e	38.5 ± 0.1 d	84.6 ± 0.0 i
MCSFA (g/100 g)	ND a	47.5 ± 0.1 h	ND a	22.5 ± 0.1 e	ND a	23.3 ± 0.1 f	15.1 ± 0.0 d	7.4 ± 0.0 c	31.0 ± 0.1 g	7.2 ± 0.0 b
LCSEFA (g/100 g)	63.6 ± 0.0 i	34.6 ± 0.0 e	7.4 ± 0.0 b	50.2 ± 0.1 a	36.5 ± 0.1 h	20.7 ± 0.0 c	35.9 ± 0.8 g	50.2 ± 0.0 a	35.1 ± 0.0 f	21.3 ± 0.0 d
SEA (g/100 g)	63.6 ± 0.0 g	82.0 ± 0.0 j	7.4 ± 0.0 a	72.7 ± 0.2 i	36.5 ± 0.1 c	44.0 ± 0.1 d	51.0 ± 0.3 e	57.6 ± 0.2 f	66.0 ± 0.1 h	28.5 ± 0.0 b
USFA (g/100 g)	36.2 ± 0.0 d	18.0 ± 0.0 a	92.6 ± 0.0 j	27.3 ± 0.2 b	63.5 ± 0.1 h	56.0 ± 0.1 g	49.0 ± 0.3 f	42.4 ± 0.2 e	34.0 ± 0.1 c	71.5 ± 0.0 i
MUFA (g/100 g)	30.8 ± 0.0 d	12.5 ± 0.0 a	63.6 ± 0.0 j	22.1 ± 0.1 b	48.1 ± 0.1 h	38.5 ± 0.1 g	36.4 ± 0.3 f	33.7 ± 0.2 e	24.6 ± 0.0 c	50.3 ± 0.0 i
PUFA (g/100 g)	5.4 ± 0.0 a	5.5 ± 0.0 a	7.4 ± 0.0 h	5.2 ± 0.2 a	15.3 ± 0.2 e	18.5 ± 0.0 f	12.6 ± 0.7 d	8.7 ± 0.4 b	9.4 ± 0.0 c	21.2 ± 0.0 g
AI <sup>d</sup>	1.7 ± 0.0 c	7.3 ± 0.0 f	0.1 ± 0.0 a	3.5 ± 0.0 e	0.5 ± 0.0 a	1.2 ± 0.0 b	1.3 ± 0.0 b	1.5 ± 0.0 bc	2.9 ± 0.0 d	0.5 ± 0.0 a

<sup>a</sup>Values are shown as means ± SDs of three replications. Means ( $n = 3$ ) with different letters in the same line are significantly different ( $P < 0.05$ ). <sup>b</sup>8:0, caprylic acid; 10:0, capric acid; 12:0, lauric acid; 14:0, myristic acid; 16:0, palmitic acid; 18:1, oleic acid; 18:2, linoleic acid; 18:3, linolenic acid. <sup>c</sup>g iodine/100g. <sup>d</sup>ND, not detectable.

**Table 3.** SPs and MPs of Palm Stearin, Coconut Oil, Canola Oil, and Their Blends before and after Chemical Interesterification<sup>a</sup>

blends	SP (°C)		MP (°C)	
	NIE	CIE	NIE	CIE
1	52.3 ± 0.1 jA	51.9 ± 0.1 iB	53.9 ± 0.2 jC	52.4 ± 0.0 jD
2	25.3 ± 0.0 cA	27.5 ± 0.2 eB	26.6 ± 0.2 cC	28.3 ± 0.0 fD
3	-6.0 ± 0.0 aA	2.0 ± 0.0 aB	-6.0 ± 0.0 aC	2.0 ± 0.0 aD
4	45.6 ± 0.3 gA	34.8 ± 0.0 fB	49.8 ± 0.2 hC	35.4 ± 0.1 gD
5	46.2 ± 0.2 hA	35.7 ± 0.1 gB	49.2 ± 0.2 gC	36.3 ± 0.1 hD
6	21.3 ± 0.3 bA	17.0 ± 0.0 bB	23.6 ± 0.1 bC	18.8 ± 0.0 bD
7	44.4 ± 0.1 fA	27.5 ± 0.1 eB	46.2 ± 0.1 fC	28.7 ± 0.0 eD
8	48.6 ± 0.1 iA	37.1 ± 0.0 hB	51.7 ± 0.0 iC	38.8 ± 0.2 iD
9	32.7 ± 0.3 dA	25.9 ± 0.1 dB	35.1 ± 0.1 dC	27.0 ± 0.1 dD
10	36.3 ± 0.1 eA	19.0 ± 0.0 cB	41.6 ± 0.4 eC	21.0 ± 0.0 cD

<sup>a</sup>Values are shown as means ± SDs of three replications. Mean ( $n = 3$ ) values with different lower case letters in the same column are significantly different ( $P < 0.05$ ). Mean ( $n = 3$ ) values with different capital letters in the same line are significantly different ( $P < 0.05$ ).

fats, candy centers, cookie fillers, nut roasting, coffee whiteners, and spray oils.<sup>6</sup>

All blends were found to contain oleic acid (greater than 12.5%) as their major unsaturated fatty acid. The content of linoleic acid in all blends was less than 22.0%, and linolenic acid was below 8.0%.

**SPs and MPs.** SPs and MPs are parameters of significant importance for characterizing and developing interesterified fats.<sup>8</sup> According to Karabulut et al.,<sup>21</sup> fat slips down the capillary tube when containing approximately 4–5% solid fat, thus enabling MP to be characterized when solid content reaches this range. The SPs and MPs of the NIE and CIE blends are shown in Table 3. Adding coconut oil and canola oil to palm stearin decreased the MP of the blends to values ranging from 51.7 to 17.0 °C.

According to Lumor et al.,<sup>22</sup> the MP of canola oil is -6 °C. The capillary tube method used in this study does not work in this temperature range, as it is performed in a water bath.

Interesterification lowered the SPs and MPs of all binary and ternary blends by reducing the proportion of high MP trisaturated triacylglycerols and increasing the percentages of disaturated–monounsaturated and monosaturated–diunsaturated triacylglycerols, which have intermediate MPs.<sup>8</sup> Other researchers have reported similar results.<sup>9–23</sup> Rousseau and Marangoni<sup>24</sup> found a directly proportional relationship between trisaturated triacylglycerols content and MP.

No significant changes in SPs and MPs of palm stearin resulting from chemical interesterification were observed (Table 3). Petrauskaitė et al.<sup>23</sup> stated that chemical interesterification of blends with high proportions of hard fat, such as palm stearin, causes only slight changes in MP. These results are similar to those obtained by Soares et al.<sup>1</sup>

Interesterified blends displayed a wide range of MPs (2.0–52.4 °C). Fats with MPs lower than body temperature can be applied directly as shortenings, because they melt completely in the mouth and produce no waxy sensation during consumption.<sup>9–21</sup> Thus, the interesterified blends 2, 3, 4, 5, 6, 7, and 9, with MPs of between 2.0 and 36.3 °C, fall within this group. In addition, blends 3 and 6 display the characteristics of liquid shortenings, which can be readily pumped and bottled at low temperatures and have MPs of between 10 and 19 °C.

Intesterified blend 8, whose MP was 38.8 °C, lies within the range of most fatty bases for producing solid and semisolid shortenings, generally represented by the all-purpose shortenings, used mainly in confectionery and bakery products and

whose representative MP is 42 °C. However, interesterified blend 1 was found to have an excessively high MP (52.4 °C) for food applications.

The coefficients for the responses determined by applying multiple regressions to the experimental data are shown in Table 4. SPs and MPs NIE blends were dependent on palm stearin, coconut oil, and canola oil and on the positive interactions between palm stearin and coconut oil, palm stearin, and canola oil and coconut oil and canola oil ( $P < 0.05$ ).

After chemical interesterification, SPs and MPs were dependent on palm stearin, coconut oil, and canola oil, on the negative interaction between palm stearin and coconut oil, and on the positive interactions between palm stearin and canola oil and coconut oil and canola oil ( $P < 0.05$ ).

**Consistency.** Figure 1 shows the consistency profiles of palm stearin, coconut oil, canola oil, and their NIE and CIE blends as a function of temperature. It was not feasible to ascertain the consistency of blend 3 because of its low MP.

The consistency of the blends decreased as a function of temperature. This decrease may be due to the gradual melting of the crystals, leading to a structurally weaker network, which is in turn responsible for the plasticity of fats.<sup>1–25</sup> At higher temperatures (20–45 °C), blends 2, 3, 4, 5, 6, 7, and 9 showed no significant differences ( $P < 0.05$ ). None of the blends exhibited measurable consistency at 50 °C NIE or at 45 °C CIE.

Consistency proved to be proportionally dependent on palm stearin concentration in the blends, before and after randomization. The increase in saturated fatty acids content of a sample strongly influences its consistency, due to their high MPs.

Overall, interesterification led to a reduction in yield values for all blends at all temperatures examined, with the sole exception of blend 2, whose consistency increased at 10, 15, 20, and 25 °C. The consistency of the interesterified blends decreased with increasing temperature, which causes the gradual melting of the crystals and consequent destruction of the crystalline network, which endows the fat with plasticity.<sup>1</sup> None of the interesterified blends exhibited measurable consistency at 45 °C.

According to Haighton,<sup>16</sup> a fat is plastic and spreadable at yield values ranging from 0.2 to 0.8 kgf/cm<sup>2</sup>. Blends 5 and 8 NIE, and the interesterified blend 5, with a yield value of 0.8 kgf/cm<sup>2</sup> at 10 °C, had satisfactory plasticity and spreadability properties for use at refrigeration temperatures, in addition to MP requisites for use in margarines, as mentioned earlier.

Table 4. Coefficients Calculated by Multiple Regression from the Experimental Results of SPs and MPs ( $P < 0.05$ )

	coefficients of multiple regression															
	$\beta_1^a$		$\beta_2^b$		$\beta_3^c$		$\beta_{12}^d$		$\beta_{13}^e$		$\beta_{23}^f$		$\beta_{123}^g$		$r^2$	
	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE
SP	52.37	51.20	25.30	28.56	-6.00	2.21	+27.40	-20.12	+92.37	+36.14	+46.57	+11.52	0.00	0.00	0.99	0.99
MP	53.97	53.12	26.63	28.71	-6.00	2.20	+38.17	-15.84	+100.93	+42.71	+53.37	+14.79	0.00	0.00	0.99	0.99

<sup>a</sup>Palm stearin. <sup>b</sup>Coconut oil. <sup>c</sup>Canola oil. <sup>d</sup>Interaction between palm stearin and coconut oil. <sup>e</sup>Interaction between palm stearin and canola oil. <sup>f</sup>Interaction between coconut oil and canola oil. <sup>g</sup>Interaction between palm stearin, coconut oil and canola oil. <sup>h</sup>Coefficient of determination.

However, there are other factors influencing the texture of a spread, such as the crystallization procedure including the variables cooling rate, degree of supercooling, mechanical working, tempering, SFC of fat used, and presence of nonfat materials.<sup>22</sup>

Between 25 and 30 °C, the interesterified blend 2 was satisfactorily spreadable (yield values between 0.8 and 1.0 kgf/cm<sup>2</sup>) but exhibited ideal plasticity at 35 °C, which is important for sensorial properties such as mouthfeel sensation and lack of adhesiveness. Thus, its consistency profile from 25 to 35 °C corroborates its suitability for application in bakery and confectionery products. The interesterified blends 4 and 8 can be classified as hard at room temperature, with a yield value of between 1.6 and 2.0 kgf/cm<sup>2</sup> in the 25–30 °C interval, which is just above the spreadability limit (1.5 kgf/cm<sup>2</sup>) at 35 °C. According to Jeyarani and Reddy,<sup>20</sup> fats considered hard at room temperature are suitable for firmer food products, where deformations must not occur during handling or stocking.

The coefficients for the responses determined by applying multiple regressions to the experimental data are shown in Table 5. For noninteresterified and interesterified blends, consistency was dependent on palm stearin, on interactions between palm stearin and coconut oil and between palm stearin and canola oil at all temperatures, and on coconut oil and on interaction between coconut oil and canola oil from 5 to 20 °C for noninteresterified blends and from 5 to 25 °C for interesterified blends ( $P < 0.05$ ). The interaction coefficients were negative for all blends, showing a eutectic interaction between palm stearin, coconut oil, and canola oil.

Eutectic behavior occurs in the blends due to the differences in the molecular size of the fatty acids and the shape and polymorph of the crystals among the three types of fat.<sup>25,26</sup> This shows that palm stearin rich in palmitic acid, coconut oil rich in lauric acid, and canola oil rich in oleic acid are incompatible with each other.

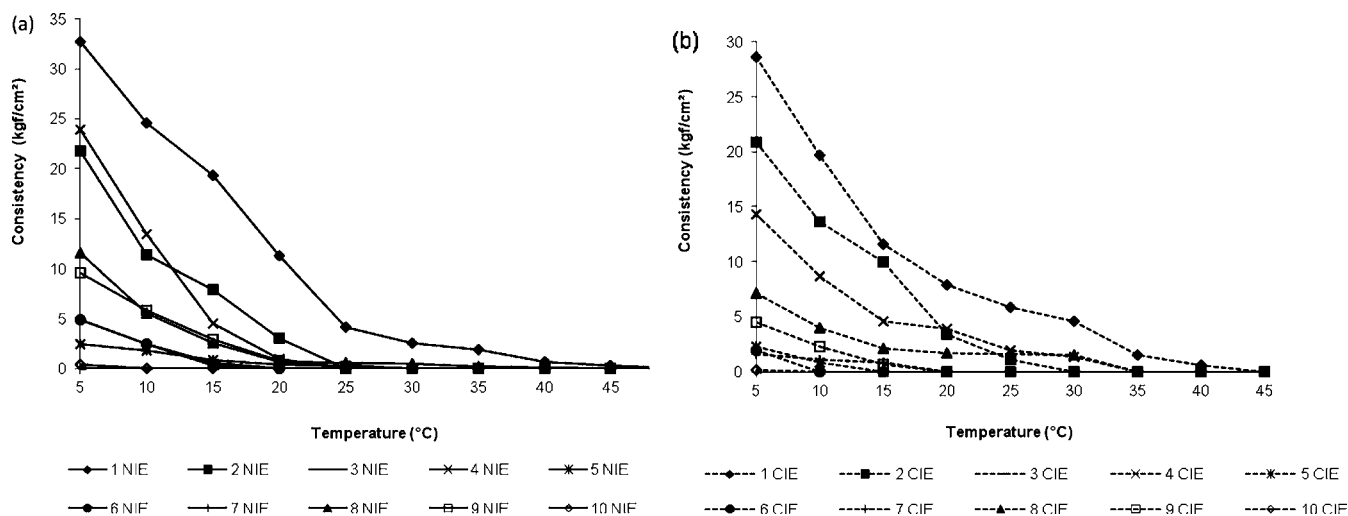
Statistical models for consistency NIE and CIE blends are shown in Figure 2 using triangular diagrams. The three vertices correspond to the responses of palm stearin, coconut oil, and canola oil. The points on the sides of the equilateral triangle represent the results of binary mixtures. The inner values indicate the responses for ternary mixtures.

The eutectic effect is clearly evidenced by depressions in the curves of consistency diagrams at 20 °C (Figure 2a,b). The higher the eutectic effect, the lower the consistency because the incompatibility among fats in solid state hinders crystallization.

**SFC.** SFC is responsible for many product characteristics in margarines, shortenings, and spreads, including their general appearance, ease of packing, spreadability, oil exudation, and organoleptic properties.<sup>27</sup>

DSC is an easy and fast technique, which is highly practical and useful for determination of SFC. At the application temperature, SFC of fats can be determined from the DSC melting curves by partial integration.<sup>28</sup> Partial areas are obtained according to the procedures described by Menard and Sichina.<sup>29</sup>

The capability of the DSC equipment enabled working at lower temperatures (down to -60 °C). Therefore, this determination was possible even in the analysis of the SFC of canola oil, which is liquid at room temperature. The SFC of palm stearin, coconut oil, canola oil, and their blends at different temperatures calculated from the DSC data are given in Figure 3a,b.



**Figure 1.** Consistency of palm stearin, coconut oil, canola oil, and their blends before (a) and after (b) chemical interesterification.

The curves show that the blends have different profiles, covering large ranges of SFC versus temperature. The SFC of the blends was proportional to the addition of coconut oil and canola oil to palm stearin, at all the temperatures analyzed.

The SFC of palm stearin was 93.9% at  $-10^{\circ}\text{C}$ , 82.3% at room temperature ( $25^{\circ}\text{C}$ ), and decreasing to 46.9% when temperature increased to  $40^{\circ}\text{C}$ . SFC obtained by DSC for palm stearin was higher than the value obtained by NMR provided by the supplier. This can be explained by the fact that the determination of SFC by DSC is done after crystallization of the samples at  $-60^{\circ}\text{C}$ , while the determination by NMR precedes crystallization at  $0^{\circ}\text{C}$ .

The SFC of palm stearin increased in the  $15\text{--}25^{\circ}\text{C}$  range and subsequently decreased. This behavior is the result of the recrystallization observed in the heating curve obtained by DSC. The results obtained were in accordance with those described by Reshma et al.<sup>30</sup> However, Alim et al.<sup>31</sup> did not report this same behavior in their samples. The recrystallization behavior was also observed in blends 8 and 5 NIE, with higher proportions of palm stearin.

The SFC of the interesterified fats was lower than their corresponding initial blends, with the exception of palm stearin between 10 and  $20^{\circ}\text{C}$ , where the recrystallization phenomenon occurred.

Under ambient temperature, coconut oil is a heterogeneous slurry of crystals admixed in liquid oil. The SFC of coconut oil was 56.9% at  $20^{\circ}\text{C}$  and 16.1% at  $25^{\circ}\text{C}$ , but at  $30^{\circ}\text{C}$ , the SFC was found to be only 0.3%, indicating that the triacylglycerols of coconut oil melted at  $25\text{--}30^{\circ}\text{C}$ . However, the SFC increased after blending with palm stearin and decreased after blending with canola oil.

The noninteresterified blend 4 had SFC similar to that found by Jeyarani, Khan and Khatoun<sup>27</sup> for a blend constituting 50% coconut oil and 50% palm stearin at  $35^{\circ}\text{C}$ . Blends 1, 4, 5, and 8 NIE were not suitable as plastic fats, because they contained excessive solids at this temperature, which may affect the mouthfeel of the product.<sup>21</sup>

SFC at room temperature ( $25^{\circ}\text{C}$ ) should be 15–35% for desirable spreadability as plastic fats.<sup>7</sup> In the case of the noninteresterified blends 7 and 9 and interesterified blends 4 and 8, SFC at room temperature ( $25^{\circ}\text{C}$ ) was within the scope of the above criteria, suggesting that blends in this study were suitable for spreadable fat or margarine stock.

Generally, SFC at  $20^{\circ}\text{C}$  corresponds to a tendency toward oil exudation and more than 10% SFC is essential to avoid oiling off.<sup>8</sup> All blends, except blend 6 NIE, had SFC higher than 10% at  $20^{\circ}\text{C}$ . Blends 3, 6, and 10 CIE had SFC lower than 10%.

According to Kim, Lumor and Akoh<sup>10</sup> SFC should be  $<32\%$  at  $10^{\circ}\text{C}$  to impart good spreadability at refrigerator temperature. The interesterified blend 10 had SFC below 32% at  $10^{\circ}\text{C}$ . Blends 3, 5, 6, 9, and 10 CIE contained SFC of 16.5, 14.6, 0.7, 18.1, and 0.3%, respectively.

For canola oil, SFC at  $-15^{\circ}\text{C}$  was 39.7% and at  $0^{\circ}\text{C}$  was 0.0%. After chemical interesterification, SFC was 32.2% at  $-15^{\circ}\text{C}$  and 18.8% at  $0^{\circ}\text{C}$ . These results are typical of liquid oils.

The coefficients for SFC after applying multiple regressions to the experimental data are shown in Table 5. SFC before chemical interesterification was dependent on palm stearin at all temperatures and on coconut oil from 5 to  $25^{\circ}\text{C}$ , on interactions between palm stearin and coconut oil and between palm stearin and canola oil at all temperatures, and between coconut oil and canola oil from 5 to  $25^{\circ}\text{C}$  ( $P < 0.05$ ). After chemical interesterification, SFC was dependent on palm stearin from 5 to  $45^{\circ}\text{C}$ , on coconut oil from 5 to  $25^{\circ}\text{C}$ , and on canola oil from 5 to  $20^{\circ}\text{C}$ , on interactions between palm stearin and coconut oil and between palm stearin and canola oil from 5 to  $45^{\circ}\text{C}$ , and between coconut oil and canola oil from 5 to  $25^{\circ}\text{C}$  ( $P < 0.05$ ). Negative interaction coefficients indicate that a eutectic interaction had occurred.

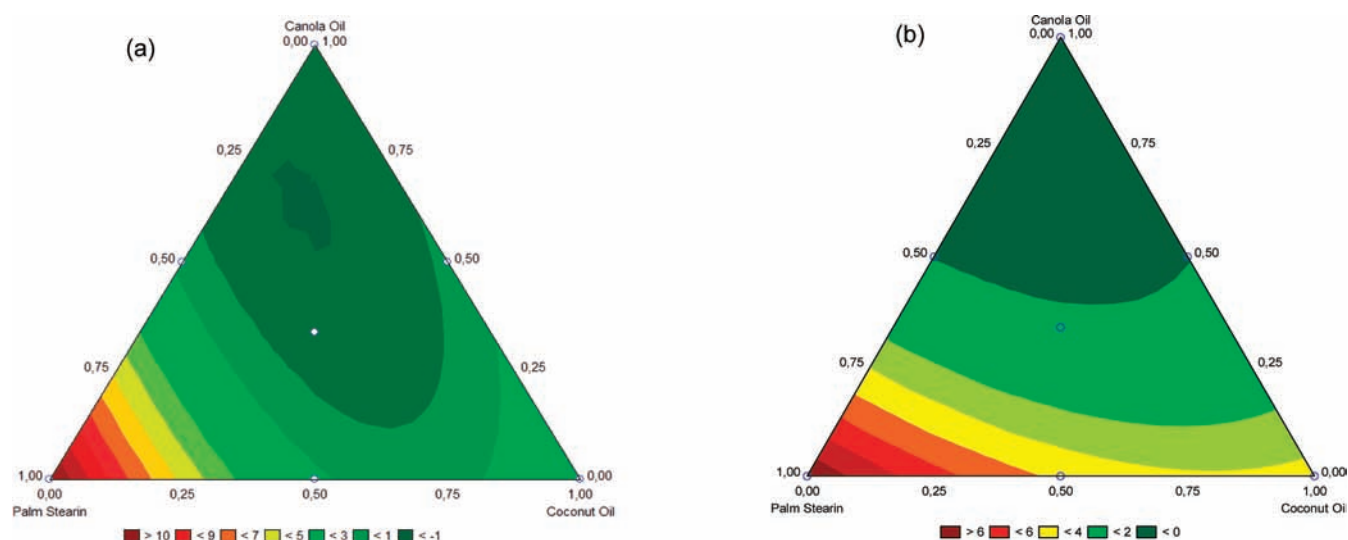
Eutectic behavior is due to the presence of residual amounts of triacylglycerols with long- and medium-chain fatty acids, respectively. This result is consistent with the findings of Norizzah et al.,<sup>8</sup> whereby interesterification eliminates or reduces eutectic interactions in a eutectic mixture. This eutectic effect is desirable if the blend is destined for use in the production of margarine and shortening.

**Validation.** ANOVA was used to verify that the proposed model adequately expresses the responses of SPs and MPs, consistency, and SFC of the NIE and CIE blends. The percentage variations explained by the models was between 90.00 and 99.99%, confirming the good fit of the regression. The low rates of residuals showed that the experimental errors were controlled and random in nature. In addition, a low rate of lack of fit and pure error was also found.

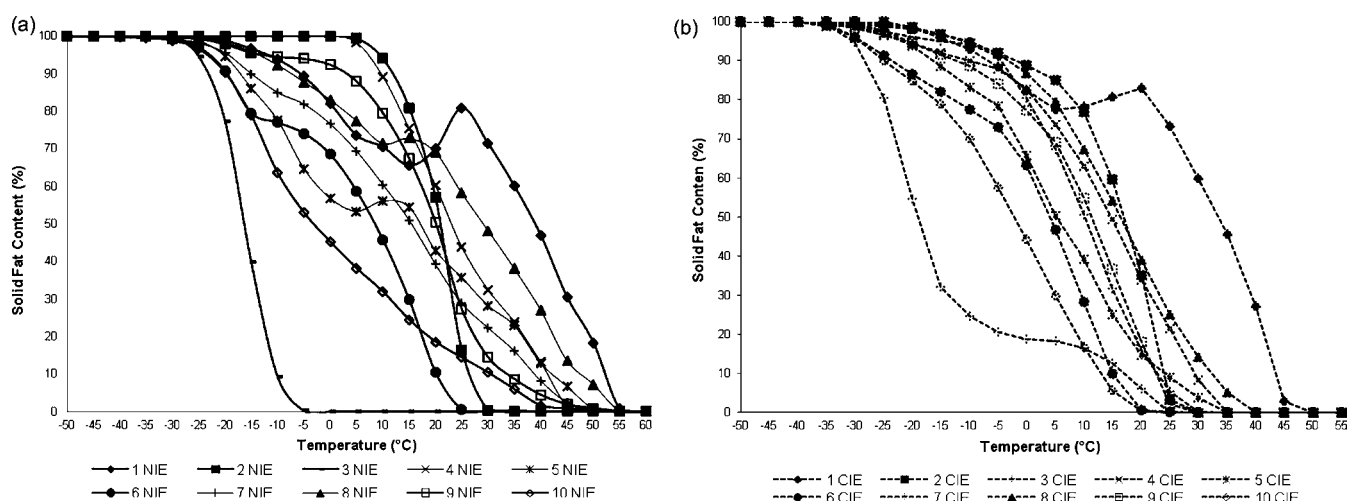
Table 5. Coefficients Calculated by Multiple Regression from the Experimental Results for Consistency and SFC ( $P < 0.05$ )

temp (°C)	$\beta_1^a$		$\beta_2^b$		$\beta_3^c$		$\beta_{12}^d$		$\beta_{13}^e$		$\beta_{23}^f$		$\beta_{123}^g$		$r^{2h}$			
	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE	NIE	CIE		
5	33.05	28.62	22.04	21.03	0.00	0.00	-17.09	-43.14	-59.17	-49.49	-27.61	-35.20	0.00	0.00	0.99	0.99		
10	24.65	19.62	11.44	13.52	0.00	0.00	-19.74	-23.65	-43.51	-21.37	-14.56	-18.96	0.00	0.00	0.99	0.94		
15	19.38	11.54	7.83	9.95	0.00	0.00	-34.99	-7.32	-33.61	-15.98	-12.95	-7.02	0.00	0.00	0.95	0.94		
20	11.18	7.96	2.98	3.41	0.00	0.00	-23.35	-7.36	-19.52	-15.91	-4.74	-7.00	0.00	0.00	0.98	0.99		
25	4.70	6.04	0.00	1.23	0.00	0.00	-7.80	-3.94	-6.59	-13.84	0.00	-4.19	0.00	0.00	0.98	0.95		
30	2.52	2.58	0.00	0.00	0.00	0.00	-4.69	-4.71	-4.78	-4.71	0.00	0.00	0.00	0.00	0.96	0.96		
35	1.87	1.95	0.00	0.00	0.00	0.00	-3.49	-3.51	-3.48	-3.51	0.00	0.00	0.00	0.00	0.97	0.97		
40	0.67	0.74	0.00	0.00	0.00	0.00	-1.25	-1.23	-1.25	-1.23	0.00	0.00	0.00	0.00	0.98	0.99		
					coefficients of multiple regression of SFC													
5	73.93	76.74	99.80	84.12	0.00	17.49	+41.08	0.00	+60.09	+5.76	+29.95	0.0	0.00	0.00	0.99	0.96		
10	65.93	77.23	94.43	75.91	0.00	15.78	+29.62	-41.52	+84.65	0.00	-13.68	-59.58	0.00	0.00	0.99	0.96		
15	70.12	79.84	81.09	34.44	0.00	11.95	0.00	-67.42	+74.07	-70.85	-46.39	-88.76	0.00	0.00	0.99	0.97		
20	80.75	82.36	56.79	34.9	0.00	5.52	-32.12	-89.10	+31.13	-108.32	-69.33	-68.25	0.00	0.00	0.99	0.99		
25	82.21	73.25	16.11	3.56	0.00	0.00	-19.01	-66.51	0.00	-109.91	-27.55	-6.30	0.00	0.00	0.99	0.99		
30	71.29	59.78	0.00	0.00	0.00	0.00	-11.70	-84.59	-7.80	-102.65	0.00	0.00	0.00	0.00	0.99	0.99		
35	59.94	45.79	0.00	0.00	0.00	0.00	-23.23	-84.39	-16.01	-84.12	0.00	0.00	0.00	0.00	0.99	0.99		
40	46.89	26.91	0.00	0.00	0.00	0.00	-40.55	-50.12	-26.01	-50.09	0.00	0.00	0.00	0.00	0.99	0.99		
45	30.52	2.78	0.00	0.00	0.00	0.00	-53.81	-5.16	-17.67	-5.13	0.00	0.00	0.00	0.00	0.99	0.98		

<sup>a</sup>Palm stearin. <sup>b</sup>Coconut oil. <sup>c</sup>Canola oil. <sup>d</sup>Interactions between palm stearin and coconut oil. <sup>e</sup>Interactions between palm stearin and canola oil. <sup>f</sup>Interactions between coconut oil and canola oil. <sup>g</sup>Interactions between palm stearin, coconut oil and canola oil. <sup>h</sup>Coefficient of determination.



**Figure 2.** Triangular diagram of consistency ( $\text{kgf}/\text{cm}^2$ ) at 20 °C of blends of palm stearin, coconut oil, and canola oil before (a) and after (b) chemical interesterification.



**Figure 3.** SFC of palm stearin, coconut oil, canola oil, and their blends before (a) and after (b) chemical interesterification.

Blends 8, 9, and 10 were used to validate the model obtained by multiple regression for the parameters SPs and MPs, consistency, and SFC. The proportion of error found for SPs and MPs, NIE and CIE blends, was lower than 20%. The proportion of errors found for SFC, before, and CIE was lower than 10% in the 5–40 °C temperature range. Errors for the consistency variable, before and after chemical interesterification, were lower than 15% at some temperatures. These percentages confirmed the predictability of the multiple regression models.

A comprehensive understanding of the functions and properties of fats or oil bases produced by interesterification is essential to outlining applications for them and obtaining food products with the desired final attributes. Chemical interesterification of palm stearin, coconut oil, and canola oil blends produced fats with different physicochemical properties. Lower consistency, SFC, and SPs and MPs were obtained, as compared with the starting blends. Therefore, palm stearin, coconut oil, and canola oil interesterified blends can be used for the production of margarine and shortenings, representing an alternative to partially hydrogenated fats.

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## ABBREVIATIONS USED

CIE, chemical interesterification and chemical interesterified blends; EIE, enzymatic interesterification; NIE, noninteresterified blends;  $x_1$ , palm stearin;  $x_2$ , coconut oil;  $x_3$ , canola oil; FAME, fatty acids methyl esters; ISO, International Organ-



ization for Standardization; AOCS, American Oil Chemists' Society; SFC, solid fat content; DSC, differential scanning calorimetry; IV, iodine value; MCSFA, medium-chain saturated fatty acids; LCSFA, long-chain saturated fatty acids; SFA, saturated fatty acids; USFA, unsaturated fatty acids; MUFA, monounsaturated fatty acids; PUFA, polyunsaturated fatty acids; AI, atherogenic index; ND, not detectable;  $\hat{y}_i$ , estimated response;  $\beta_j$ , coefficients estimated by the least-squares method;  $x_i$ , dependent variables; MP, melting point; SP, softening point; NMR, nuclear magnetic resonance; ANOVA, analysis of variance;  $\beta_1$ , palm stearin;  $\beta_2$ , coconut oil;  $\beta_3$ , canola oil;  $\beta_{12}$ , interaction between palm stearin and coconut oil;  $\beta_{13}$ , interaction between palm stearin and canola oil;  $\beta_{23}$ , interaction between coconut oil and canola oil;  $\beta_{123}$ , interaction between palm stearin, coconut oil, and canola oil;  $r^2$ , coefficient of determination

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