Composition, Physical and Textural Characteristics of Soft (Tub) Margarines

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Soft (tub) margarines were analyzed for fatty acid, *trans* and polyunsaturated fatty acid (PUFA) content. Soybean and sunflower-palm kernel-palm margarines contained high levels of *cis-cis* methylene interrupted (CCMI)-PUFA. Canola and canola-palm products contained the lowest amounts of saturated fatty acids. Polymorphic forms of the crystals were as follows: soybean beta prime, canola beta, canola-palm and a sunflower-palm kernel-palm—a mixture of beta and beta prime. Dropping points of the fats ranged from 27.3 to 34.2°C. Softening points of the products were higher especially for margarines that existed in the beta form. Texture was determined by cone penetrometer, constant speed compression and penetration. Soybean margarines were generally most resistant to deformation. The solid fat content (SFC) of the "whole" margarines as determined by the Bruker Minispec was found to be slightly lower than that of the separated fat (AOCS-method) at 10°C. Correlation of values within the textural methods was significant ($P<.01$), but not between the texture and SFC of the product which means that the nature of the crystal network also plays a role in texture.

KEY WORDS: Fatty acids, melting point, polymorphism, polyunsaturated fatty acids, soft margarine, solid fat, texture, *trans* fatty acids.

Soft (tub) margarines have replaced stick margarines to a great extent especially in Canada. In the United States stick margarines are still popular partly because the stick margarines in that country are of softer consistency. Postmus *et al.* (1) analyzed the chemical composition and deMan *et al.* (2) the physical and textural composition of a series of North-American stick margarines. They found that stick margarines made of canola oil, the oil frequently used in Canada for margarine manufacture, were harder in texture than the United States soybean-containing margarines. Canola margarines require the incorporation of palm oil in order to stabilize the beta-prime crystal structure. Yap *et al.* (3) demonstrated that a slightly hydrogenated palm oil at a 10% level was more effective than unhydrogenated palm oil at the same level, but unhydrogenated palm oil at a 15% level was more effective than at a 10% level.

This study was undertaken to evaluate a series of soft margarines.

MATERIALS AND METHODS

Samples of different brands were purchased from supermarkets in the provinces of Ontario and Quebec, and the state of Ohio in the United States. They were transported in coolers containing ice and stored at refrigerator temperature (4°C) thereafter. Sample 2 was a product identified as "whipped," i.e. gas was incorporated. Sample 3 was labeled "low calorie," i.e. low fat, high moisture content. The fat was obtained by melting part of the margarines in the oven. After removal of the water layer, the fat was dried and filtered.

Fatty acids. The fatty acid composition of the fat was determined by transesterification with Na-methoxide and analyses of the methyl esters by gas liquid chromatography (4) using a Shimadzu GC-8A gas chromatograph with a 2 m glass column packed with 10% SP-2330 on 100/120 chromosorb WAW (Supelco, Bellefonte, PA) and operated at 170°C. *Trans* fatty acid content was determined by infra-red spectroscopy on the methyl esters according to AOCS Method Cd 14-61 (5) by using a Beckman model 4300IR spectrophotometer. The methyl esters in this case were prepared according to AOCS Method Ce 2-66 (5). *Cis-cis* methylene interrupted polyunsaturated fatty acids (CCMI-PUFA) were determined enzymatically with lipoxygenase (6).

Fat crystal structure. The polymorphic forms of the crystals in the products were established by X-ray diffraction by using a 601 Diffractis X-ray generator and a Guinnier X-ray diffraction camera, Model FR 552 (Enraf Nonius, Delft, The Netherlands) which was operated at 10°C (7). Crystal size was visualized by polarized light microscopy by using an Olympus model BH polarizing microscope. Fat crystal structure was determined within two days of purchase.

Softening and dropping points. The softening points of the products and the dropping points of the separated fats were determined with the Mettler FP 80 Central processor, using a heating rate of 1°C/min. For the determination of the dropping point, the fat was melted and then solidified in the cups at −10°C for 1 hr (8). In the softening point determination the sample cup was filled by pushing it directly into the product. Excess product was carefully removed.

Texture. All measurements were carried out in a 5°C cold room. A cone penetrometer as described by AOCS Method Cc 16-60 was used to determine hardness. The hardness index (g/mm) was calculated by dividing the mass of the cone assembly (92.5 g) by the depth of penetration in mm (9).

Constant speed penetration and compression measurements were carried out by means of the Ottawa Texture Measurement System (OTMS) using a 2-kg load cell which was hooked up to a strain-gage conditioner and an Apple IIe computer as previously described (2). In the penetration test, samples were contained in small cups (25 × 16 mm diam.). A stainless-steel punch (.332 cm^2) was driven into the sample. In the compression test, cylindrical samples (20 × 20 mm diam.) were compressed to 10 mm (2).

Solid fat content (SFC) of the samples was measured by pulsed nuclear magnetic resonance (p-NMR), by using a Bruker PC/20 Series NMR analyzer (Minispec). The SFC of the separated fat was determined by using AOCS Method Cd 10-57 with a tempering step at 25°C instead

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TABLE 1

Fatty Acid Composition of Soft Margarines

Sample	16:0	18:0	18:1	18:2	18:3	*trans*	Saturates + *trans*	CCMI[a] PUFA
Soybean								
United States								
1.	10.8	7.4	46.6	31.7	2.6	11.7	30.3	30.6
2.	10.7	7.0	45.3	33.1	2.7	12.4	30.5	27.0
3.	10.1	6.6	39.1	38.3	5.0	10.3	27.4	42.3
Canada								
4.	10.4	6.9	38.0	37.5	6.1	12.8	30.6	42.0
5.	10.6	7.3	38.9	36.4	5.7	14.1	32.4	36.5
6.	10.0	6.7	40.7	35.8	5.6	14.1	31.3	38.1
7.	9.5	6.8	42.6	34.5	5.5	13.7	30.5	32.0
8.	9.7	6.4	42.8	34.7	5.5	14.1	30.7	35.7
9.	10.3	5.9	38.3	38.5	5.9	12.6	29.2	41.7
Canola								
10.	4.4	6.3	66.0	14.8	6.1	12.7	23.8	18.8
11.	4.1	8.5	68.6	12.0	4.1	17.8	30.8	11.0
Canola & palm								
12.	6.2	5.7	62.3	17.8	5.5	13.1	25.8	20.9
13.	5.9	5.8	63.7	16.0	6.3	9.4	21.7	19.4
14.	5.7	5.7	62.6	16.8	6.9	8.5	20.4	22.0
Sunflower-palm kernel-palm								
15.[b]	9.2	9.4	17.7	56.9	0.5	0.6	24.9	52.8
16.[c]	12.2	4.9	20.5	57.0	0.8	1.0	22.2	54.9

[a]*cis-cis* — methylene interrupted polyunsaturated fatty acids.

[b]Also 3.8% of 12:0 and 1.3% of 14:0.

[c]Also 2.6% of 12:0 and 1.0% of 14:0.

of 26.7°C (5). SFC in the margarines was determined as previously described (10).

Texture measurements and SFC of product were performed within one week of purchase. All analyses were run in duplicate with the exception of solid fat content which was run in triplicate, compression and constant speed penetration in quadruplicate and hardness measurements which were the means of six readings. Correlation coefficients were obtained by linear regression analysis.

RESULTS

The fatty acid composition of the soft margarines is displayed in Table 1. The margarines were divided into the following groups: soybean, canola, canola-palm, and sunflower-palm kernel-palm. Soybean oil contains approximately 10.5% of palmitic acid, canola 4.5% and palm oil 44%. The level of palm oil addition to canola margarines is not so high that mixtures of canola-palm cannot be distinguished from soybean margarines. The sunflower-palm kernel-palm margarines had labels that specified the individual oils.

The soybean margarines contained more CCMI-PUFA than the canola margarines, but the total saturates were lower in the canola and canola-palm samples (Table 1). When the *trans* and saturate contents were added, the canola-palm mixtures contained lower amounts than the soybean samples. The sunflower-palm kernel-palm mixtures contained the highest amount of CCMI-PUFA of all samples (Table 1). Their *trans* content was very low. The label of sample 15 indicated that the palm kernel oil (PKO) was hydrogenated and that of sample 16 modified. PKO is low in unsaturation; it has an iodine value of 15 (11). Hydrogenation would result in complete or nearly complete saturation. Judging from the 12:0 and 14:0 fatty acid content (Table 1), the fat in sample 15 contained 8% hydrogenated palm kernel oil and sample 16 about 6%. By using the percentage of sunflower oil in the margarines listed on the labels, the calculated sunflower oil content in the fats was 85 and 87.5%, respectively. The remainder of the fat in these margarines was declared on the label of sample 15 as hydrogenated and of sample 16 as modified palm oil. Modified palm and palm kernel oil could either mean fractionated, hydrogenated or interesterified.

Compared with stick margarines, as analyzed by Postmus *et al.* (1), the soybean, canola, and canola palm soft margarines contained more 18:2 and less saturates + *trans* fatty acids.

The fat crystals of the soybean margarines were in the beta prime form, the canola-only margarines in the beta form and the canola-palm margarines contained a mixture of beta and beta prime crystals (Table 2). Stick margarines containing only canola also were found by Postmus *et al.* (1) to be in the beta form; canola-palm stick margarines were either in the beta or beta prime form depending on the amount of palm oil incorporation or whether the palm oil was slightly hydrogenated. Incorporation of slightly hydrogenated palm oil resulted in a more stable product than unhydrogenated palm oil (1,3). The estimated palm oil addition in samples 12, 13 and 14 was 4 to 5%, insufficient to keep the crystals in the beta prime form. Higher amounts of palm oil will result in a more stable product and in addition may permit the

TABLE 2

Dropping Points of Fats of Soft Margarine, Softening Points of Soft Margarines (as is) and Polymorphic Form of Crystals of Soft Margarines

Sample	Dropping point °C	Softening point °C	Polymorphic form[a]
Soybean			
United States			
1.	29.4	32.6	β′
2.[b]	30.0	34.5	β′
3.[c]	30.4	40.2	β′
Canada			
4.	32.7	34.9	β′
5.	34.2	35.5	β′
6.	30.3	31.5	β′
7.	31.4	32.0	β′
8.	32.9	32.2	β′
9.	29.3	30.9	β′
Canola			
10.	27.3	34.1	β
11.	31.4	35.3	β
Canola-palm			
12.	31.1	32.4	β=β′
13.	31.6	33.1	β′>>>β
14.	31.2	32.8	β′=β
Sunflower-palm kernel-palm			
15.	33.0	33.6	β′
16.	29.5	35.2	β′=β

[a]Relative amounts of the two forms.

[b]Whipped margarine.

[c]Low calorie margarine.

canola oil to be hydrogenated to a lesser degree or permit an increase of liquid oil content, thereby increasing the CCMI-PUFA. Sample 16 also contained a mixture of beta prime and beta crystals. Samples of this brand-name margarine were examined for their polymorphic form on several occasions during the year with the same results. Possible reasons for sample 16 being less stable in the beta prime polymorphic form than sample 15 could be: i) sample 16 contained less PKO, ii) sample 16 contained fractionated palm stearin (FPS) instead of hydrogenated palm oil, iii) sample 16 contained palm oil or FPS that was interesterified. FPS was found by Yap *et al.* (12) to be less stable in the beta prime form than palm oil, which in turn was less stable than hydrogenated palm oil. PKO contains approximately 8% of palmitic acid (16:0), palm oil 44% and FPS 60% (depending on the fractionation process used). Sample 16 contained more palmitic acid than sample 15 (Table 1). Further analysis of the solid fraction of the fat of these samples is required to explain their difference in polymorphic behavior.

The mean dropping point of all the soft margarines in Table 2 was 31.0°C, whereas that of 22 all-vegetable stick margarines in a previous study was 34.9°C (1). The softening points (Table 2) were higher than the dropping points. The difference between the softening points and dropping points was more pronounced in the margarines containing only beta crystals (samples 10 and 11) and sample 16. Postmus *et al.* (1) also found a greater difference between softening points and dropping points in stick margarines containing beta crystals than those containing beta prime crystals. In the dropping point determination, the fat is frozen at −10°C and crystallizes in the beta prime form. The beta forms have higher melting points than the beta prime polymorphic forms. The fat of sample 16 contained the lowest amount of solids at 10°C of all samples in Table 4. It has been our experience that fats which are low in solids content are sometimes difficult to crystallize, which could account for the large difference between dropping point and softening point in sample 16. Sample 2 was a whipped margarine and the incorporated gas could have slowed down the flow of the melted fat in the sample cup, while in the low calorie sample 3 (45% fat) the emulsified water phase could have caused the same phenomenon, resulting in the higher than usual softening points.

The canola margarines containing only beta crystals had no surface sheen and appeared dull. Their crystals were large as seen through the polarizing light microscope. The crystals of the remaining margarines were small including the margarines containing beta and beta prime crystals.

The textural attributes at 5°C are displayed in Table 3. Mean values for hardness, penetration and compression for the soybean soft margarines (Samples 1 and 4-9, Table 3) were 8.5, 6.5 and 1.56, respectively, while those for the canola margarines (Samples 10-14) were 7.3, 4.7 and 1.13. The canola-palm and the sunflower-palm kernel-palm products (Samples 15 and 16) were slightly softer than the rest of the products, with the exclusion of soybean 9. In a previous study by deMan *et al.* (2) mean values for hardness, penetration and compression for soybean stick margarines at 10°C were 15.6, 11.0 and 2.5 and

TABLE 3

Textural Characteristics of Soft Margarines at 5°C

Sample	Constant weight—cone penetrometer g/mm	Constant speed	
		Penetration N/cm²	Compression N/cm²
Soybean			
United States			
1.	8.9	8.1	1.50
2.	8.8	6.3	1.66
3.	7.0	4.2	1.15
Canada			
4.	8.3	6.0	1.43
5.	9.4	6.9	1.85
6.	10.4	7.2	2.01
7.	8.4	6.9	1.50
8.	9.4	7.5	2.01
9.	4.8	2.7	.61
Canola			
10.	8.1	3.9	.89
11.	10.5	7.5	1.85
Canola-palm			
12.	6.0	3.9	1.11
13.	6.5	4.5	.92
14.	5.6	3.6	.89
Sunflower-palm kernel-palm			
15.	6.9	5.4	1.05
16.	6.1	5.1	.70

TABLE 4

Solid Fat Content by p-NMR (%) of Fat Separated from Soft Margarines and of the Actual Soft Margarines

	Soft margarines		Separated fat			
Sample	5°C	10°C	10°C	20°C	25°C	30°C
Soybean						
United States						
1.	13.9	12.6	13.2	7.9	4.4	2.0
2.	13.4	12.1	12.1	5.4	4.8	2.2
3.	8.5	7.9	12.9	6.9	3.7	2.3
Canada						
4.	15.9	13.2	14.9	8.8	6.1	3.5
5.	16.5	14.3	17.2	11.7	7.8	3.9
6.	16.1	14.9	16.3	9.0	6.0	1.9
7.	15.2	13.9	15.6	9.1	5.1	2.9
8.	16.8	15.6	16.1	9.9	7.7	4.3
9.	14.1	12.5	14.5	8.5	5.0	1.9
Canola						
10.	12.9	12.2	14.2	7.6	5.0	3.0
11.	16.9	14.7	18.3	9.9	7.2	4.1
Canola						
12.	15.2	13.9	14.0	8.5	5.2	3.1
13.	13.1	12.4	13.5	8.5	6.3	3.2
14.	11.6	11.2	12.0	7.4	5.6	2.6
Sunflower-palm kernel-palm						
15.	11.4	9.9	10.4	6.7	5.9	3.0
16.	9.7	8.5	7.1	4.5	3.5	1.6

TABLE 5

Correlation Coefficients Within Textural Methods and Between Textural Methods and SFC of Product at 5°C

Method	r
Hardness and penetration	.877
Hardness and compression	.916
Penetration and compression	.873
Hardness and SFC-product	.655
Penetration and SFC-product	.486
Compression and SFC-product	.789

for canola stick, 17.1, 16.0 and 5.3, respectively. The canola stick margarines were significantly harder in the compression test than the soybean stick margarines. This was not the case with the soft margarines analyzed in this study. Stick margarines have a more extensive crystal network because of their higher solid content than soft margarines. The compression test is a sensitive method in evaluating the strength of the crystal network (2).

Generally, the soft margarines at 5°C were softer than stick margarines at 10°C which were analyzed in a previous study (2). Some of these stick margarines, especially the corn and sunflower products, came close in textural attributes at 10°C to those of the soft margarines at 5°C (2).

The SFC of the fats and that of the products is presented in Table 4. The mean SFC in the product at 10°C was 12.8% and that of the fat was 14.0%. Similar differences between the SFC of margarine products and that of the separated fat were obtained in stick margarines (2). It is possible that the water present in the margarine interferes with the measurement of the SFC. The direct mode was used in the Bruker Minispec which measures the ratio of the number of hydrogen nuclei in the solids to the total number of hydrogen nuclei. The solid fat content of sample 3 when measured at 10°C on the original product was much lower than the value obtained at the same temperature on the separated fat. This may have been caused by the high water content of this product. Samples 15 and 16 contained the lowest amounts of solids in the group analyzed (Table 4). The correlation between SFC of the product at 10°C and that of the fat at 10°C was significant ($P<.01$). Sample 3 was excluded because of its high water content. Correlations within the textural methods were significant ($P<.01$) (Table 5). Samples 2 and 3 were excluded when running correlation coefficients because the products were different from the rest of the samples. The correlation between peak force compression and SFC of the product also was significant ($P<.05$). Correlations between hardness index and penetration and that of SFC were not significant, which means that the nature of the crystal network also plays a role in the texture of the products.

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