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IMPROVED SUCROSE ESTERS IN BREADMAKING[1]

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ABSTRACT Cereal Chemistry 53(5): 615-626

Several commercial sucrose esters of various hydrophile-lipophile balances (HLB) were compared in breadmaking as replacements of wheat-flour lipids and 3% shortening. The beneficial effect of sucrose esters on loaf volume and texture when baked with petroleum ether (PE) defatted flour increased with increased HLB values. Sucrose ester with an HLB of 1.0 only partially replaced flour lipids, but with an HLB of 8.0, it was a better replacement than the one with an HLB of 1.0, and two sucrose esters with an HLB of 14.0 functionally replaced both flour lipids and 3% shortening. Removing the PE-soluble fractions enhanced the beneficial effects of the two sucrose esters with an HLB of 14.0. Bake tests and thin-layer chromatography showed that the most effective components of the sucrose esters were those with R_f values close to those of digalactosyl diglycerides of flour lipids.

Recently, lipid-related surfactants have received increased attention by researchers in the field of baking. Certain surfactants not only improve shelf-life as antistaling agents (1–3) and modify the rheological properties of dough (4–6) but also counteract the adverse effects on loaf volume and texture of protein-rich additives used for producing high-protein breads (7–11). Also, shortening can be replaced by some surfactants in breadmaking (9,12,13), and wheat-flour lipids can be partially replaced by surfactants at binding sites of dough constituents during dough formation (6,14).

We studied the effects on baking properties of replacing wheat-flour lipids with several commercial sucrose esters which were mixtures of mono-, di-, and triesters and varied in hydrophile-lipophile balance (HLB). Sucrose esters were separated into petroleum ether (PE)-soluble and PE-insoluble fractions. The fractions were characterized by thin-layer chromatography, and the characterized components were compared with flour lipid components and standard lipids.

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MATERIALS AND METHODS

Materials

Regional Baking Standard (RBS-74)—an untreated, straight-grade flour—was experimentally milled (Allis) from a composite grist of many wheat varieties grown at locations throughout the Great Plains in 1973. The flour contained 12.4% protein (N × 5.7) and 0.50% ash (14% mb), and had good loaf-volume potential and medium mixing and oxidation requirements.

Commercial sucrose esters were manufactured by Dai-Ichi Kogyo Seiyaku Co., Ltd. and Dai-Nippon Sugar Manufacturing Co., Ltd., both companies in Tokyo, Japan. Sucrose esters have been approved for general food use in Japan and for certain food uses in several European countries. They have not yet been cleared by the U.S. Food and Drug Administration.

Reference lipids were from Applied Science Laboratories, Inc., State College, Pa.

Organic solvents were analytical reagent grade, and solutions were prepared from analytical reagent-grade compounds.

Analytical Procedures

Protein (macro and micro), ash, and moisture contents were determined as described in Method 46-11, Method 46-13, Method 08-01, and Method 44-15A, respectively, of the AACC Approved Methods (15). The baking procedure described by Finney and Barmore (16–18) and Finney (19), and adapted by Shogren *et al.* (20) for 10 g (14% mb) flour was used. The breadmaking formula was the same as described by Shogren *et al.* (20), except that 10 ppm potassium bromate and 100 ppm ascorbic acid were used as oxidizing agents. Dry ingredients (flour, nonfat milk solids, and sucrose esters) were blended in a Stein Mill for 30 sec. The amount of sucrose ester added was 80 mg/10 g flour (0.8%). Shortening was a commercial product of vegetable origin, partly hydrogenated, mp 41° C. The standard deviation for the average of triplicated loaf volumes was 1.43 cc. Loaf volume was determined by dwarf rapeseed displacement immediately after the bread was taken from the oven. After cooling, loaves were cut and their crumb grains and textures were evaluated. This code was employed: S, satisfactory; Q, questionable; and U, unsatisfactory.

Defatting Flour

Lipids were extracted from 1 kg RBS-74 flour with about 2.8 l. PE (bp 30° to 60° C) for 48 hr in a large Soxhlet extraction apparatus. Solvent of the lipid extract was evaporated at reduced pressure below 30° C and the lipid was stored at −18° C. The defatted flour was air-dried until the odor of PE was not detected, then stored at 4° C.

Fractionating Sucrose Esters

Three grams of sucrose ester was wrapped with filter paper and placed in a cellulose extraction thimble (Whatman, 33 × 80 mm). PE-solubles were extracted with 175 ml PE in a medium Soxhlet extraction apparatus for 16 hr. Condensation rate was 2 to 3 drops per sec. The PE-soluble fraction was evaporated at reduced pressure below 30° C, and the PE-insoluble fraction was air-dried until the odor of solvent was gone. Three to eight lots of PE-solubles

were combined and pulverized in a mortar to a product of uniform particle size.

Unfractionated sucrose esters and their PE-soluble and PE-insoluble fractions are denoted as UF, SF, and IF, respectively.

Thin-Layer Chromatography (tlc)

Glass plates (20 × 20 cm) were coated with a 250-μm layer of silica gel G, and the thin layers were activated for 3 hr at 130°C. The solvent systems for one-dimensional ascending development were: hexane-diethylether-methanol (80:20:1, v/v/v, solvent system I) for standard nonpolar lipids, flour lipids, and PE-soluble and PE-insoluble fractions of sucrose esters; and chloroform-methanol-water (65:25:4, v/v/v, solvent system II) for standard polar lipids, flour lipids, and sucrose ester fractions. Plates were sprayed with a 0.6% $K_2Cr_2O_7$ solution in 55% H_2SO_4 and heated for 25 min at 180°C (21). The plates were

TABLE I
Properties of Commercial Sucrose Esters[a]

Type of Sucrose Esters	Monoester Content[b] %	HLB Value[b]	Petroleum Ether Solubles %
F-10	1.2	1.0	85.5
F-70	41.0	8.0	27.5
F-160	71.2	14.0	4.2
P-1570	70.0	14.0	2.3

[a]F-10, F-70, and F-160 were from Dai-Ichi Kogyo Seiyaku Co., Ltd., Tokyo, Japan. P-1570 was from Dai-Nippon Sugar Manufacturing Co., Ltd., Tokyo, Japan.
[b]Manufacturers' data.

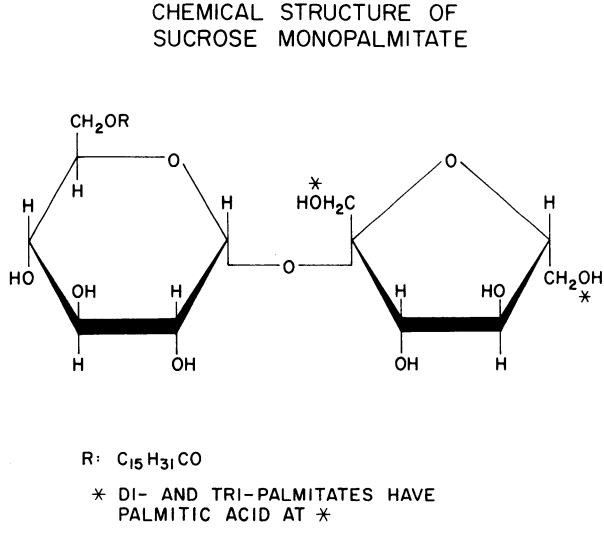

Fig. 1. Chemical structure of sucrose monopalmitate.

photographed under ultraviolet light. Specific sprays for glycolipids were 0.2% α-naphthol in ethanol followed by a light spray with 95% H_2SO_4 (22) and diphenylamine (23).

RESULTS

Characteristics of Sucrose Esters

Effectiveness of a surfactant for a particular application depends, in part, on its HLB. The higher the HLB number, the greater its affinity for water and, conversely, the lower the HLB number, the greater its affinity for oil (24,25).

Sucrose esters contain hydrophilic (hydroxy) and lipophilic (alkyl) groups as shown in Fig. 1. The HLB value increases as monoester content of sucrose ester increases (Table I). As di- and triester content decreases, more free hydroxy radicals of sucrose are available and the sucrose ester becomes more hydrophilic.

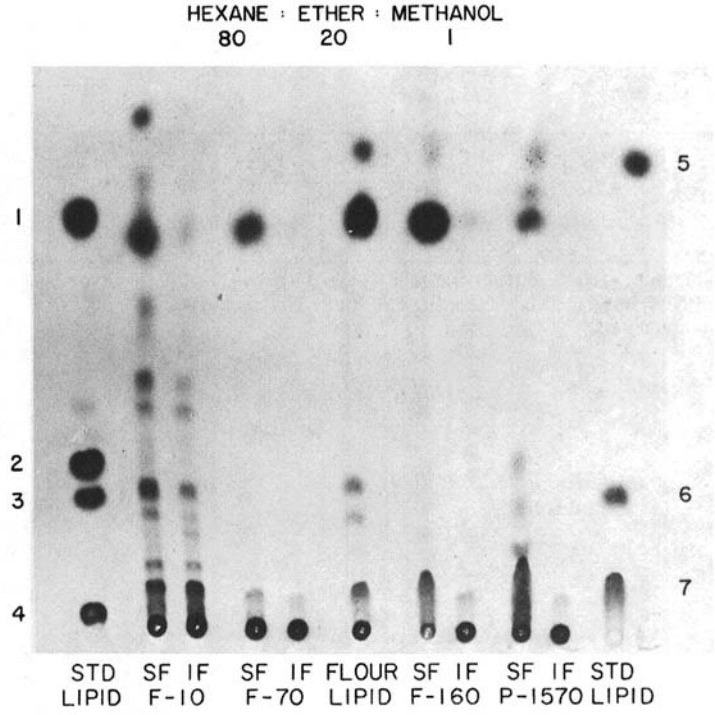

Fig. 2. Thin-layer chromatogram of standard lipids, sucrose ester fractions, and wheat-flour lipids developed with hexane-diethyl ether-methanol (80:20:1), charred with a 0.6% $K_2Cr_2O_7$ solution in 55% H_2SO_4, and photographed under uv light. From left to right, standard lipids, PE-soluble (SF) and PE-insoluble (IF) fractions of F-10 and F-70, wheat-flour lipids, SF and IF fractions of F-160 and P-1570, and standard lipids. Standard lipids were: 1) triolein; 2) 1,3 diolein; 3) 1,2 diolein; 4) monoolein; 5) β-sitosteryl palmitate; 6) β-sitosterol; and 7) oleic acid. Amount applied: 15 μg of each standard lipid and 100 μg of flour lipids and of each sucrose ester fraction.

Consequently, the PE-soluble fraction of a sucrose ester decreases with increasing HLB value. Petroleum ether, with a low polarity, is a good solvent for highly lipophilic compounds. Although F-160 and P-1570 have the same HLB value, their monoester contents and amounts of PE-solubles differ slightly. The company technical bulletin gives fatty acid components of P-1570 as 30% stearate and 70% palmitate, determined by gas chromatography. Most of the sucrose esters were white to creamy powders. PE-soluble fractions of F-160 (F-160-SF) and of P-1570 (P-1570-SF) were waxy. No N was detected in 30-mg samples analyzed by the micro-Kjeldahl method (15).

Characterizing Sucrose Esters by Thin-Layer Chromatography (tlc)

The PE-soluble fractions of sucrose esters contained mainly nonpolar components, which were separated by solvent system I and the PE-insoluble fractions, except for F-10-IF, contained few nonpolar components (Fig. 2). The major components of PE-soluble fractions of sucrose esters seemed to be triglycerides and free fatty acids. Diglycerides were absent in F-70-SF and F-160-

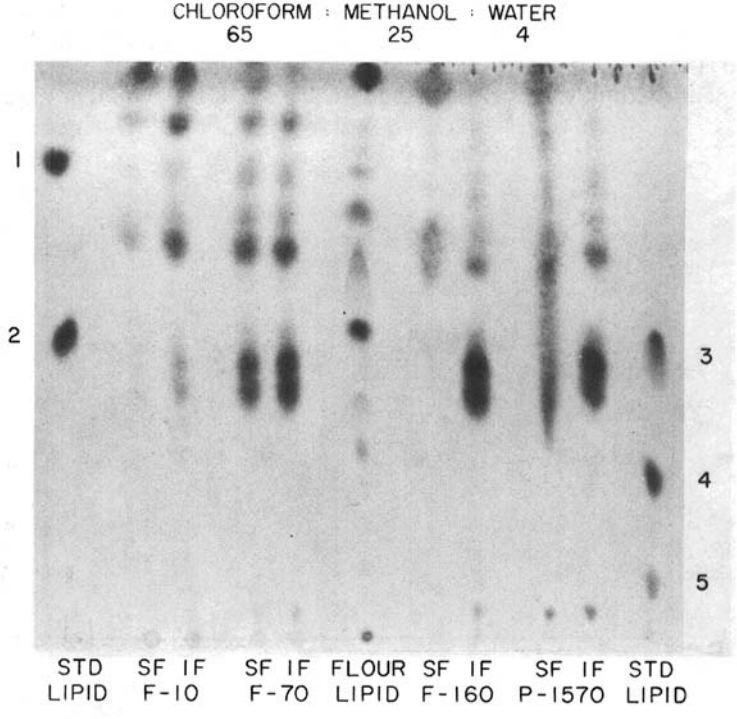

Fig. 3. Thin-layer chromatogram of standard lipids, sucrose ester fractions, and wheat-flour lipids developed with chloroform-methanol-water (65:25:4), charred with a 0.6% $K_2Cr_2O_7$ solution in 55% H_2SO_4, and photographed under uv light. The legend of Fig. 3 is the same as in Fig. 2 except that the standard lipids were: 1) monogalactosyl diglyceride, 2) digalactosyl diglyceride, 3) phosphatidylethanolamine, 4) phosphatidylcholine, and 5) lysophosphatidylcholine.

SF. Identifications of glycerides and fatty acids in Fig. 2 are based only on R_f values of compounds.

Major components of the PE-insoluble fractions of sucrose esters (Fig. 3) had R_f values similar to those of digalactosyl diglyceride (DGDG) and phosphatidylethanolamine (PEA). The next most abundant components of the PE-insoluble fractions had R_f values between those of monogalactosyl diglyceride (MGDG) and DGDG. The chromatograms of both PE-soluble and PE-insoluble fractions of F-70, whose monoester content was 41.0%, were similar for separations by solvent system II. However, as compared to F-70-IF, F-70-SF contained a substantially larger amount of triglycerides and a smaller amount of components with R_f values close to those of DGDG and PEA.

When the developed thin-layer plates were sprayed with diphenylamine solution (23), supposedly specific for glycolipids, and heated at 100° C for about 30–40 min, yellow spots appeared on a blue background. However, the spray also stained standard phospholipids yellow. Thus, we found diphenylamine solution rather nonspecific and insensitive. Spraying plates with 0.2% α-naphthol solution (22) stained glycolipids purple (Fig. 4). Standard and flour glycolipids turned red-purple; sucrose esters turned gray-purple. Nonpolar components except three of F-10-SF developed in solvent system I gave no color reaction with α-naphthol. Those three unidentified components had R_f values between those of standard tri- and diglycerides and stained faint purple. Components with R_f values close to the R_f value of DGDG would be monoesters,

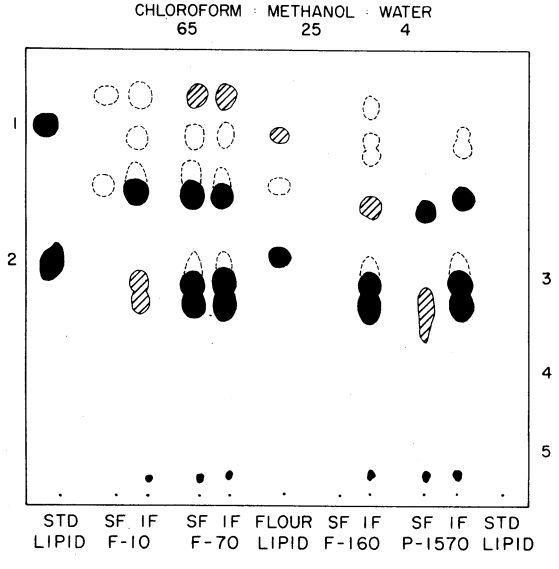

Fig. 4. Thin-layer chromatogram of standard lipids, sucrose ester fractions, and wheat-flour lipids developed with chloroform-methanol-water (65:25:4). Plate was sprayed with a 0.2% α-naphthol solution in ethanol, followed by light spray with 95% H_2SO_4 and heating for 30 min at 120° C. Red-purple to gray-purple spots are sketched. The darker the shading of spots, the higher the intensity of color. The legend of Fig. 4 is as described in Fig. 3. Standard phospholipids 3, 4, and 5 gave no colored spots.

those with R_f values between the R_f values of DGDG and MGDG would be diesters, and those with R_f values higher than the R_f of MGDG would be triesters. The chromatograms of F-10-UF and F-10-SF were similar; as were those of F-70-UF, F-70-SF, and F-70-IF; those of F-160-UF and F-160-IF; and those of P-1570-UF and P-1570-IF.

Sucrose Esters in Breadmaking

Defatting flour decreased dough absorption about the same amount as omitting shortening increased it (Table II). Flour PE-extract was 0.8%. Adding 0.8% sucrose esters to defatted flour increased water absorption in both shortening and shortening-free doughs (average increases of 0.9 and 2.4%, respectively, above absorption of defatted flour). Similar results were reported by Pomeranz *et al.* (7) for doughs containing untreated flour and soy flour, with 0.5% added sucrose esters. There was little variation in water absorption of doughs containing the different fractions of the same sucrose esters.

Mixing times of defatted flours with added shortening were somewhat shorter, and without added shortening slightly longer, in general, than the corresponding mixing times of untreated flours (Table II).

With 3% shortening, defatted RBS-74 flour gave a loaf volume of only 65.0 cc compared to 75.5 cc for the untreated flour (Table III). When shortening was omitted, however, loaf volumes for both untreated (65.5 cc) and defatted (62.0

TABLE II
Absorption and Mixing Characteristics of Defatted RBS-74 Flour with Various Sucrose Esters with or without Shortening

Type of Sucrose Esters Added[a]		Shortening Added		No Shortening Added	
		Baking abs. %	Mixing time min	Baking abs. %	Mixing time min
Untreated RBS-74 Flour					
None		63.8	3-7/8	65.3	3-1/2
Defatted RBS-74 Flour					
None		62.5	3-3/4	63.0	3-3/4
F-10	UF	63.0	3-5/8	65.0	3-5/8
	SF	63.0	3-1/2	65.0	3-1/2
	IF	63.0	3-3/4	65.0	3-7/8
F-70	UF	64.0	3-5/8	66.0	3-3/8
	SF	63.5	3-5/8	66.0	3-3/8
	IF	64.0	3-5/8	66.0	3-5/8
F-160	UF	63.5	3-1/2	64.5	3-1/2
	SF	63.5	3-1/2	64.0	3-3/8
	IF	63.5	3-5/8	65.0	3-5/8
P-1570	UF	63.5	3-7/8	66.0	3-3/4
	SF	63.5	3-1/2	66.0	3-1/2
	IF	63.5	3-5/8	66.5	3-1/2

[a]UF = unfractionated sucrose ester; SF = PE-soluble fraction; and IF = PE-insoluble fraction.

cc) flours were small and distinctly unsatisfactory. Although the addition of the unfractionated sucrose esters or their fractions, except F-160-SF and P-1570-SF in the no-shortening series, increased loaf volume, only the unfractionated sucrose esters F-160 and P-1570 and their PE-insoluble fractions gave loaf volumes about equal to loaf volume of the untreated control with shortening. Loaf volume increased with increasing monoester content of sucrose esters with or without shortening. This finding agrees with that of Finney and Shogren (26) on a sugar-free formula for high-protein breads.

Neither fraction of highly lipophilic F-10, with or without shortening added, effectively replaced the native flour lipids and restored loaf volume. F-70, with an intermediate HLB value, was more beneficial than F-10. For loaves made with shortening, F-70-UF gave a loaf volume nearly equal to that of the untreated control flour. Also, the highly hydrophilic, unfractionated esters F-160 and P-1570 and their PE-insoluble fractions gave loaf volumes much larger than the loaf volume of untreated RBS-74 with shortening. In the no-shortening baking series, F-160-UF, F-160-IF, and P-1570-IF gave loaf volumes at least as large as the loaf volume of the untreated control with shortening. Adding the PE-soluble fractions of both F-160 and P-1570 without shortening gave loaf volumes even smaller than the loaf volume of the defatted control (62.0 cc). Thus, F-160-SF

TABLE III
Loaf Volume and Crumb Grain of Bread Baked with 10 g Defatted RBS-74 Flour and 80 mg Sucrose Esters with or without Shortening

Type of Sucrose Esters Added[a]		Shortening Added		No Shortening Added	
		Loaf volume cc	Crumb grain[b]	Loaf volume cc	Crumb grain[b]
Untreated RBS-74 Flour					
None		75.5	S	65.5	Q
Defatted RBS-74 Flour					
None		65.0	Q	62.0	Q-S
F-10	UF	69.5	S	63.5	Q-U
	SF	67.0	S	62.5	Q-U
	IF	68.5	S	64.5	Q-U
F-70	UF	74.5	S	64.0	Q-U
	SF	71.0	S	64.5	Q-S
	IF	71.5	S	66.0	Q
F-160	UF	80.5	S	75.5	S
	SF	71.5	Q-S	55.0	Q-U
	IF	80.5	S	77.0	S
P-1570	UF	77.5	S	70.0	Q
	SF	70.5	S	59.5	Q-U
	IF	79.5	S	77.0	S

[a]UF = unfractionated sucrose ester; SF = PE-soluble fraction; and IF = PE-insoluble fraction.
[b]S = satisfactory; Q = questionable; U = unsatisfactory.

and P-1570-SF were detrimental to breadmaking; the detrimental effect was largely counteracted by the addition of shortening.

Bread crumb grains of loaves made with the defatted flour without shortening generally were distinctly inferior, except when F-160-UF, F-160-IF, and P-1570-IF were added. Thus, F-160-UF, F-160-IF, and P-1570-IF could effectively replace both native flour lipids and 3% shortening.

Typical examples of microloaves (10 g flour) described in Table III are shown in Figs. 5 and 6.

GENERAL DISCUSSION

Adding the unfractionated sucrose esters to defatted flour improved baking characteristics, with or without shortening. The beneficial effect of unfractionated sucrose esters on loaf volume of defatted flour, with or without shortening, increased with increasing HLB values. The improving effect of sucrose esters with high HLB values was enhanced by removal of their PE-soluble fractions. Removing the PE-soluble fraction from F-10 (low HLB value) had no improving effect; and fractionation of F-10 is impractical because of high content

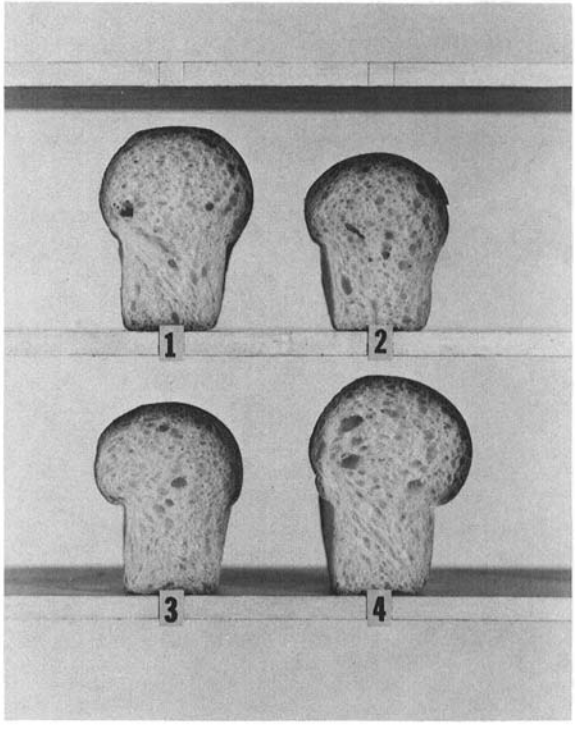

Fig. 5. Microloaves (10 g flour) baked with 3% shortening from: 1) untreated flour, 2) defatted flour, 3) defatted flour + unfractionated F-10, and 4) defatted flour + unfractionated F-160.

of PE-soluble fraction. Superiority of unfractionated F-70 probably was due to complementary effects of PE-soluble and PE-insoluble fractions in the presence of shortening. Without shortening, F-70-IF was superior to F-70-UF or F-70-SF. However, because of the limited effect of F-70-IF as a replacement for flour lipids and 3% shortening, the value of fractionating F-70 is questionable. Sucrose esters F-160 and P-1570, with high HLB values, functionally replaced both the natural flour lipids and 3% shortening. Bake test and tlc showed that the most effective components of the sucrose esters were those with R_f values close to those of DGDG of flour lipids.

Recently, there has been a substantial improvement in functional (breadmaking) properties of commercially available sucrose esters. For example, as much as 4% sucrose tallowate previously was required to restore breadmaking quality of high-protein breads (8). Now, it is possible to obtain acceptable high-protein breads with as little as 0.5% sucrose esters (26). The effect on loaf volume of as little as 0.15 to 0.25% of the presently available sucrose esters added to wheat flour alone is fully equivalent to that of 3% shortening (26). On the other hand, adding previously available sucrose tallowate to unsupplemented wheat flour even had deleterious effects on loaf volume and crumb grain (27).

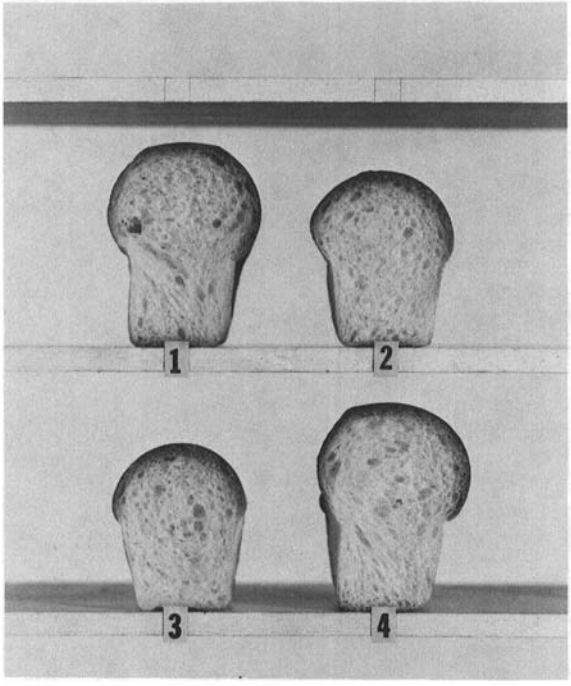

Fig. 6. Microloaves (10 g flour) baked from: 1) untreated flour + 3% shortening, 2) defatted flour, 3) defatted flour + PE-soluble fraction of F-160, and 4) defatted flour + PE-insoluble fraction of F-160.

The present study emphasizes that the effectiveness of sucrose esters is proportional to their monoester contents. Also it shows that their effectiveness can be further enhanced by removal of the undesirable nonpolar and other components together with, presumably, di- and triester fractions with nonpolar solvent.

Acknowledgment

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