Improved Synthesis of Sucrose Fatty Acid Monoesters

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ABSTRACT: The base-catalyzed synthesis of four sucrose fatty acid esters (caprylate, laurate, myristate, and palmitate) was performed in dimethylsulfoxide by transesterification of sucrose with the corresponding vinyl esters using disodium hydrogen phosphate as catalyst. In using a molar ratio sucrose/vinyl ester 4:1 and mild reaction conditions (40°C and atmospheric pressure), yields were higher than 85%. The isolated sucroesters had a higher percentage of monoesters (≥90%) and a lower content of diesters in comparison with commercial derivatives. In all cases, 2-*O*-acylsucrose was the major product (≥60%) in the monoester fraction.

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KEY WORDS: Acylation, fatty acid ester, nonionic surfactant, organic solvents, sucrose monoester, transesterification, vinyl ester.

Sucrose fatty acid esters are nonionic surfactants that may be produced from renewable, inexpensive, and readily available resources (1). They are widely used in foods, cosmetics, and pharmaceuticals (2,3). Apart from their emulsifying properties, they are completely biodegradable, harmless to the environment, nontoxic, skin-compatible, odorless and tasteless, and are digested as a blend of sucrose and fatty acids in the stomach (4).

A wide range of hydrophilic-lipophilic balance (HLB) values can be attained with these products. This balance can be modulated by varying (i) the alkyl chain length of the acyl groups, (ii) the number of ester groups per molecule, or (iii) the degree of unsaturation of the acyl chains (5). The properties of a sucrose ester can range from those of a water-soluble surfactant (high HLB) to an oil-soluble emulsifier (low HLB). Mono-, di- and triesters have extensive use as emulsifiers, especially those with fatty acids containing 12 or more carbon atoms (6).

Current industrial synthesis of sucroesters involves high temperature, reduced pressure, anhydrous conditions, and/or expensive catalysts. Furthermore, none of the current industrial processes is particularly selective. They all afford mixtures of compounds differing in their degree of esterification and/or the position of acylation (7). Although for many applications these mixtures of mono-, di-, and triesters are convenient, chemical reactions that preferentially produce sucrose monoesters in a single and economical procedure may represent an attractive approach to new uses. Derivatives containing three or more fatty acid residues are very hydrophobic and of limited application. For this reason, new methods oriented to achieve a higher selectivity in the reaction or to provide economical purification procedures are being developed (1). The selective synthesis of monoesters may also be useful in studying the functional properties of single components of commercial derivatives (8).

The selective monoacylation of sucrose is difficult to achieve because of the similar reactivity of the eight hydroxyl groups and to the existence of intramolecular migration processes (9). Recently, we developed a simple procedure for the regioselective acylation of sucrose using vinyl laurate and an inexpensive basic catalyst (10). In the present work, we report the preparation of a homologous series of sucrose esters with *n*-acyl chains of 8, 12, 14, and 16 carbon atoms, their chemical characterization, and the comparison of their monoester content vs. commercially available sucrose monoesters.

EXPERIMENTAL PROCEDURES

Chemicals. Sucrose and dimethylsulfoxide (DMSO) were supplied by Merck (Darmstadt, Germany). Anhydrous disodium hydrogen phosphate was purchased from Panreac (Barcelona, Spain). Vinyl laurate was obtained from Fluka (Buchs, Switzerland); vinyl caprylate, vinyl myristate, and vinyl palmitate were from TCI (Tokyo, Japan). Molecular sieves (3 Å, 8–12 mesh) and orcinol/ferric chloride (Bial's reagent) were from Sigma (St. Louis, MO). Of the commercial sucrose esters, sucrose laurate was purchased from Fluka, sucrose caprate and palmitate from Sigma, and sucrose caprylate from Calbiochem (La Jolla, CA). Sucrose laurate L70-C was kindly donated by Sisterna (Roosendad, The Netherlands), and Ryoto® sucrose esters L-1695, L-595, M-1695, P-1670, P-1570, P- and S-1670 were from Mitsubishi-Kagaku Foods Co. (Tokyo, Japan). All other reagents and solvents were of the highest available purity and used as purchased.

Thin-layer chromatography (TLC). TLC was performed on silica gel 60 F_{254} plates (Merck) with chloroform/methanol 4:1 (vol/vol) as eluent; spots were detected by dipping the plates into a 20% solution of Bial's reagent in ethanol, drying, and heating at 120ºC for 5 min.

Gas–liquid chromatography (GC). GC was used to follow transesterification of sucrose with vinyl caprylate and for analysis of caprylate esters. Analyses were performed with a Varian 3400CX instrument (Varian Chromatography Systems, Sugar Land, TX) fitted with a (15 m × 0.25 mm) 5% diphenyl-

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silicone column (Sugelabor S.A., Madrid, Spain) using flame-ionization detection. Injector temperature was 290ºC and detector temperature was 300ºC. Helium was used as carrier gas. The temperature program was: 180ºC initial temperature, held for 1 min, then increased to 240ºC at 30ºC/min, then increased to 280ºC at 15ºC/min, then increased to 320ºC at 5ºC/min and held for 10 min at 320ºC. All samples were subjected to pre-column derivatization with 1-(trimethylsilyl)imidazole (Fluka) according to Sweeley *et al.* (11).

High-performance liquid chromatography (HPLC). HPLC was carried out using a system equipped with an SP 8810 pump (Spectra-Physics Inc., San Jose, CA), a Nucleosil 100-C18 column (250 × 4.6 mm, Análisis Vínicos S.L., Tomelloso, Spain), maintained at 40ºC, and a Shodex refractive-index detector SE-61 (Showa Denka, Tokyo, Japan). Integration was performed using the Varian Star 4.0 software. HPLC was used to follow acylation of sucrose with vinyl laurate, myristate, and palmitate, and for analysis of the composition of the corresponding esters. Analysis of the ratio of monoesters/diesters used a mobile phase composed of methanol and water 95:5 (vol/vol) with a 1.1 mL/min flow rate. The separation of sucrose monoesters (regioisomers) was achieved with a methanol/water 7:1 (vol/vol) as the mobile phase at a flow rate of 1.1 mL/min.

Proton nuclear magnetic resonance spectroscopy (^1H NMR). ^{1}H NMR was performed to characterize the major components of the reaction mixtures. Spectra were recorded on a Varian (Palo Alto, CA) INOVA (300 MHz) spectrometer at 30ºC for solutions in CD_3OD. Chemical shifts are referred to the methanol multiplet, centered at 3.50 ppm. Spectroscopy data for the major components of reaction of sucrose with vinyl laurate are summarized below. The ^{1}H NMR spectra of the corresponding octanoyl, myristoyl, and palmitoyl derivatives showed chemical shifts and coupling constants within ±0.05 ppm and ±0.3 Hz, respectively, of those found for the lauroyl compounds.

Synthesis of sucrose monoesters. Initially, esterification of sucrose was performed on an analytical scale using 0.58 mmol of sucrose and 1 mL of DMSO (dried over molecular sieves for 5–7 d before use) with vinyl laurate as the acylating agent at different molar ratios, in the presence of anhydrous disodium hydrogen phosphate (100 mg) as catalyst. Reactions were carried out at several temperatures. Aliquots were withdrawn at different times, centrifuged using 0.45-µm filters, and analyzed by TLC and/or HPLC (data not shown).

After establishing the best reaction conditions (on the basis of higher conversion to monoester in the shorter time), the transesterification was performed on a large scale with vinyl caprylate (C_8), laurate (C_{12}), myristate (C_{14}), and palmitate (C_{16}). Reactions were found to be significantly reproducible. In a typical procedure, sucrose (20 g) was dissolved in DMSO (100 mL) and anhydrous disodium hydrogen phosphate (10 g) was added. The mixture was stirred at 40ºC for 15 min. After addition of the corresponding vinyl fatty acid ester (15 mmol), the reaction was allowed to proceed, at the same temperature, until the vinyl ester was almost completely transformed. All reactions were monitored by TLC and GC (for caprylate esters) or HPLC (for laurate, myristate, and palmitate esters). Aliquots for analysis were taken in duplicate.

Once the reaction had been completed, the reaction mixture was left to cool to room temperature (5–10 min), and *n*-hexane (100 mL) was added. The mixture was vigorously stirred and then cooled to –20ºC. The *n*-hexane, which took up the residual vinyl esters and crystallized the disodium hydrogen phosphate, was decanted. The DMSO was allowed to warm at room temperature (15–20 min) and then filtered. The solid was washed with 1-butanol (5 mL), to recover pure catalyst. The liquid phase, containing sucrose esters and sucrose, was mixed with water (100 mL) and extracted with cyclohexane/1-butanol 1:1 (vol/vol) (3 × 200 mL). The organic phases were pooled and washed with a saturated sucrose solution diluted 1:1 (vol/vol) with water (2 × 50 mL), in order to eliminate the residual DMSO, and solvents were evaporated off.

Sucrose monoesters were further purified from the crude residue by the addition of ethyl acetate (250 mL, preheated to 40ºC) with vigorous stirring until dissolution was completed, filtration of the remaining sucrose, and concentration of the filtrate by evaporation of solvent. The solution was allowed to stand while cooling; the precipitated monoesters were then filtered off and dried under vacuum.

Monoesters were analyzed by GC (C_8) or HPLC (C_{12}–C_{16}), and major components were characterized by ^{1}H NMR. Spectral data for lauroyl monoesters are given as examples as follows.

2-*O*-Lauroylsucrose (δ, ppm): 5.53 (*d*, 1H, $J_{1,2}$ = 3.8 Hz, H-1), 4.59 (*dd*, 1H, $J_{2,3}$ = 10.1 Hz, H-2), 4.16 (*d*, 1H, $J_{3',4'}$ = 8.0 Hz, H-3′), 4.00 (*t*, 1H, $J_{4',5'}$ = 8.0 Hz, H-4′), 3.87 (*t*, 1H, $J_{2,3} = J_{3,4}$ = 10.0 Hz, H-3), *ca.* 3.85 (1H, H-6a), *ca.* 3.80 (*m*, 2H, H-5′), *ca.* 3.74 (*m*, 2H, H-6′a + H-6′b), *ca.* 3.72 (*m*, 1H, H-5), *ca.* 3.68 (*m*, 1H, H-6b), 3.51 (*d*, 1H, $J_{1'a,1'b}$ = 11.8 Hz, H-1′a), 3.41 (*dd*, 1H, $J_{4,5}$ = 9.0 Hz, H-4), 3.37 (*d*, 1H, H-1′b), 2.39 (*m*, 2H, *J* = 7.4 Hz, –C*H*$_2$–CO–), 1.63 (*m*, 2H, –C*H*$_2$–CH$_2$–CO–), 1.28 (*m*, 16H, –CH$_2$– chain), 0.89 (*t*, 3H, *J* = 7.0 Hz, CH$_3$–).

3-*O*-Lauroylsucrose (δ, ppm): 5.43 (*d*, 1H, $J_{1,2}$ = 3.8 Hz, H-1), 5.21 (*t*, 1H, $J_{2,3}$ = 10.0 Hz; $J_{3,4}$ = 9.6 Hz, H-3), 4.10 (*d*, 1H, $J_{3',4'}$ = 8.0 Hz, H-3′), 4.02 (*t*, 1H, $J_{4',5'}$ = 7.6 Hz, H-4′), 3.91 (*m*, 1H, $J_{5,6a}$ = 1.8 Hz, $J_{5,6b}$ = 6.0 Hz, H-5), *ca.* 3.80 (*m*, 1H, H-5′), *ca.* 3.76 (*m*, 4H, H-6a + H-6b + H-6′a + H-6′b), 3.64 (*d*, 1H, $J_{1'a,1'b}$ = 12.2 Hz, H-1′a), 3.60 (*d*, 1H, H-1′b), 3.57 (*dd*, 1H, $J_{2,3}$ = 10.0 Hz, H-2), 3.50 (*t*, 1H, $J_{4,5}$ = 9.7 Hz, H-4).

RESULTS AND DISCUSSION

Synthesis of sucrose monoesters. Sucrose monoesters were synthesized from unprotected sucrose in DMSO, using vinyl esters as acylating agents and an alkaline catalyst (disodium hydrogen phosphate). Unlike other transesterification reactions employing alkyl fatty acid esters, those involving vinyl esters are not limited by competition of the alcohol released, since vinyl alcohol formed during the process tautomerizes to

the low-boiling-point acetaldehyde (i.e., the equilibrium is shifted toward the ester formation) (12). This permits the use of less drastic conditions, avoiding the use of reduced pressure and high temperatures.

Acylation of sucrose was performed in dry DMSO, in which sucrose is very soluble (880 g/L at 40ºC) (13). Although substantially anhydrous conditions were not necessary, DMSO was dried over molecular sieves for 5–7 d before use, as even small amounts of moisture may change the rate of reaction.

Initially, several experimental variables were tested, such as temperature, amount of catalyst and molar ratio of reactants, using vinyl laurate as acylating agent (data not shown). We observed by TLC that the reaction could be performed at a temperature from ambient to reflux. The optimal temperature was in the range 40 to 45ºC. At higher temperatures, although the acylation proceeded faster, the monoester was partially transformed to diesters, and the presence of degraded sucrose was observed.

The influence of the amount of catalyst was also studied by TLC. It was varied between 0.5 and 100 mg/mL. As expected, the higher the amount of Na_2HPO_4, the faster the process.

Furthermore, the influence of the molar ratio of sucrose/vinyl laurate was analyzed. As shown in Figure 1, if the reaction is carried out by adding four moles of sucrose per mole of vinyl laurate, monoesters are obtained quantitatively in a short time (5 h). However, under equimolar conditions or using a fourfold excess of vinyl laurate with respect to sucrose (Fig. 2), diesters are formed after the initial stages of the reaction.

Scaleup of this process under the best reaction conditions (molar ratio of sucrose/vinyl ester 4:1, 40ºC, atmospheric pressure, 100 mg/mL of Na_2HPO_4) was performed with vinyl caprylate, laurate, myristate, and palmitate.

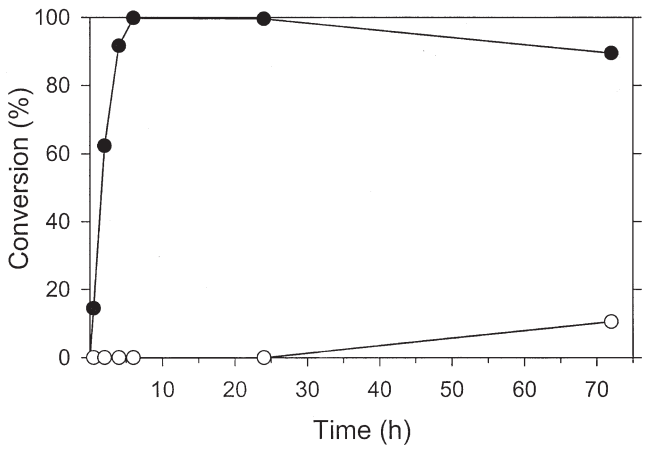

FIG. 1. Effect of the molar ratio of sucrose/vinyl laurate in the course of acylation. The initial concentration of sucrose was 0.60 M, and the concentration of vinyl laurate was 0.15 M. The formation of monoesters (●) and diesters (○) was determined by high-performance liquid chromatography (HPLC). The conversion is referred to the percentage of the initial vinyl laurate transformed into monoesters and diesters. Conditions: 40°C, 100 mg/mL Na_2HPO_4.

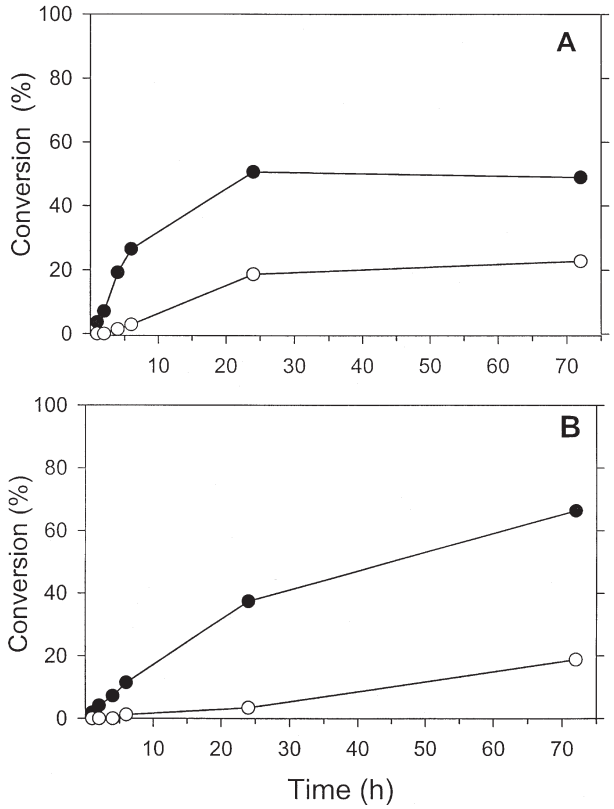

FIG. 2. Effect of the molar ratio of sucrose/vinyl laurate in the course of acylation. The initial concentration of sucrose was 0.60 M, and the following concentrations of vinyl laurate were used: (A) 0.60 M; (B) 2.4 M. The formation of monoesters (●) and diesters (○) was determined by HPLC. The conversion is referred to the percentage of the initial sucrose transformed into mono- and diesters. Conditions: 40°C, 100 mg/mL Na_2HPO_4. For abbreviation see Figure 1.

The monoesters were isolated from the reaction medium by a quite simple procedure. At the end of the synthesis, the reaction was stopped by treatment with hexane, which takes up the residual vinyl ester and precipitates the catalyst, thus allowing their reutilization. Without this extraction, reactions could progress during the following purification steps. The resulting DMSO solution was then diluted with water and extracted with a mixture of cyclohexane and 1-butanol. This extraction mixture ensures the total recovery of sucrose esters that remain dissolved in the organic phase. Residual DMSO was removed from the organic phase by washing with an aqueous sucrose solution. Evaporation of solvent afforded a crude residue, which contained the sucrose esters and a considerable amount of sucrose (≥10% w/w, as determined by GC or HPLC). Although there are applications for such mixtures, a further purification was performed by recrystallization in ethyl acetate. Yields and operating conditions are summarized in Table 1. When using vinyl palmitate or stearate, the acylation proceeded one order of magnitude slower than

TABLE 1
Synthesis of Sucrose Esters in Dimethylsulfoxide[a]

Acyl donor	Reaction time (h)	Conversion[b] (%)	Yield[c] (%)	2-*O*-Acylsucrose[d] (%)
Vinyl caprylate	3	90	86	59.8
Vinyl laurate	5	99	97	72.4
Vinyl myristate	40	94	90	67.2
Vinyl palmitate	40	92	87	69.8

[a]Experimental conditions: 0.6 M sucrose, 0.15 M vinyl fatty acid ester, 100 mg/mL Na_2HPO_4, 40°C.
[b]Based on the weight of residual vinyl fatty acid ester.
[c]Based on vinyl fatty acid ester as limiting reagent and assuming product is entirely monoester.
[d]Referred to the percentage of 2-*O*-acylsucrose in the monoester fraction, determined by gas–liquid chromatography (GC) (sucrose caprylate) or high-performance liquid chromatography (HPLC) (sucrose laurate, myristate, and palmitate).

with caprylic or lauric esters (see Table 1). Nevertheless, reactions were nearly quantitative in all cases.

Composition of isolated sucrose esters. The products obtained were analyzed by GC (sucrose caprylate) or HPLC (sucrose laurate, myristate, and palmitate). Figure 3 shows the chromatograms obtained and those corresponding to the commercially available sucrose laurates L70-C and L-1695, obtained under conditions for separation of monoesters from diesters, although the different regioisomers were not completely defined. As illustrated in Figure 3, our derivatives were substantially monoesters, with a monoester/diester ratio and purity higher than the commercial compounds.

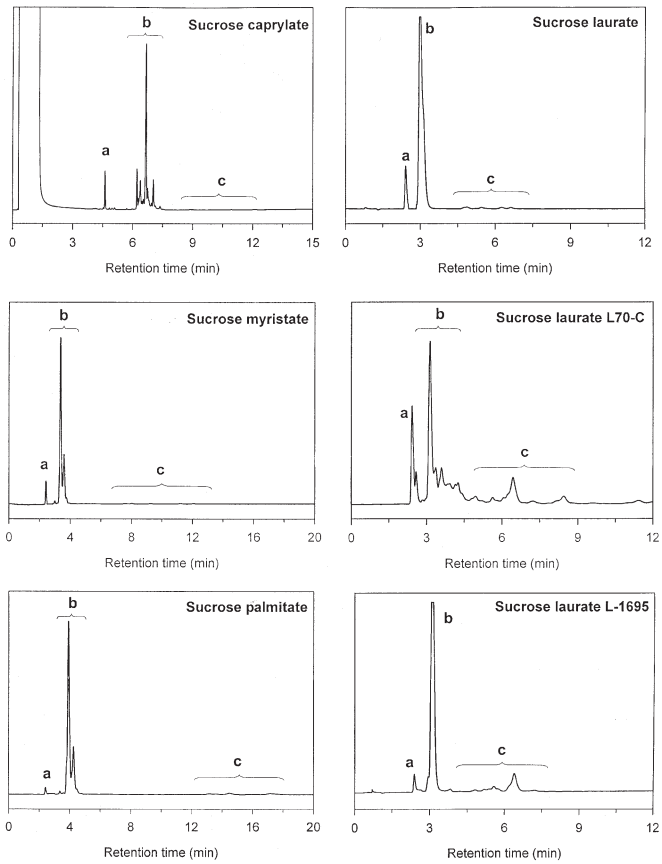

FIG. 3. Chromatographic analysis of synthesized sucrose esters [sucrose caprylate, by gas–liquid chromatography (GC); sucrose laurate, myristate, and palmitate, by HPLC] and commercial sucrose laurates [L70-C from Sisterna (Roosendad, The Netherlands) and L-1695 from Mitsubishi-Kagaku] under conditions to analyze the ratio monoester/diester. The peaks corresponding to sucrose (a), monoesters (b), and diesters (c) are indicated. Analytical conditions are described in the Experimental Procedures section. For abbreviations see Figure 1.

Table 2 summarizes the weight composition of our derivatives and a number of commercial sucroesters used in food or cosmetics, or sold as standards. Compared with the Ryoto commercial compounds from Mitsubishi-Kagaku [one of the main manufacturers of sucroesters in the world (3)], the esters isolated in this work contain a lower percentage of diesters [less than 4% (w/w) for caprylate, laurate and myristate, and 9% for palmitate].

Figure 4 shows the chromatograms obtained in conditions for separation of positional monoesters. The reaction was selective for certain hydroxyl groups. ^{1}H NMR analyses of the monoesters (see Experimental Procedures section) showed that 2-*O*-acylsucroses were the major compounds in the four products, in the percentages given in Table 1. This contrasts with most of the commercial samples, where the primary 6- and 6′-monoesters are the main products.

An explanation for the regioselectivity found may be the catalyst-induced activation of the most acidic hydroxyl of sucrose (the 2-OH of the glucose moiety) to a more nucleophilic alkoxide. In this context, Chauvin *et al.* (14) reported the syn-

TABLE 2
Composition (by weight) of Commercial and Synthesized Sucrose Esters

Compound	Source[a]	Composition (% w/w)[b] Sucrose	Monoesters	Diesters
Sucrose caprylate	Calbiochem	2	95	3
Sucrose caprylate	This work	7	92	1
Sucrose caprate	Sigma	5	93	2
Sucrose laurate	Fluka	2	95	3
Sucrose laurate L70-C[c]	Sisterna	11	50	39
Sucrose laurate L-1695	Mitsubishi-Kagaku	2	77	21
Sucrose laurate L-595[d]	Mitsubishi-Kagaku	0	27	43
Sucrose laurate	This work	5	91	4
Sucrose myristate M-1695	Mitsubishi-Kagaku	1	77	22
Sucrose myristate	This work	4	94	2
Sucrose palmitate P-1670[e]	Mitsubishi-Kagaku	0	64	22
Sucrose palmitate P-1570[e]	Mitsubishi-Kagaku	0	59	29
Sucrose palmitate P-	Mitsubishi-Kagaku	2	88	10
Sucrose palmitate	Sigma	9	65	26
Sucrose palmitate	This work	1	90	9
Sucrose stearate S-1670[f]	Mitsubishi-Kagaku	0	54	20

[a]Calbiochem (LaJolla, CA); Sigma (St. Luis, MO); Sisterna (Roosendad, The Netherlands); Mitsubishi-Kagaku Foods Co. (Tokyo, Japan).
[b]Estimated by GC for sucrose caprylate and by HPLC for the rest of the compounds (see Experimental Procedures section).
[c]This compound is a complex mixture of sucrose esters of different fatty acids (mostly lauric acid).
[d]Contains approximately 30% of higher esters.
[e]P-1670 and P-1570 contain approximately 14 and 12% of sucrose monostearate, respectively.
[f]Contains 26% of sucrose monopalmitate. See Table 1 for abbreviations.

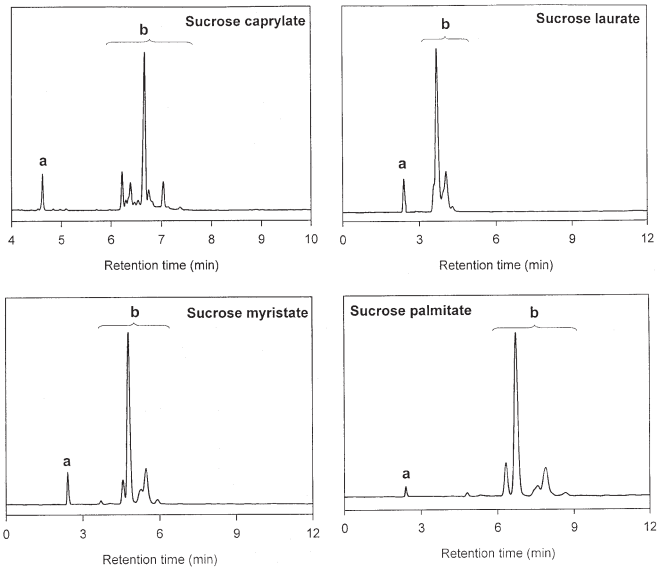

FIG. 4. Chromatographic analysis of synthesized sucrose esters (sucrose caprylate, by GC; sucrose laurate, myristate, and palmitate, by HPLC) under conditions to separate the different positional isomers. The peaks corresponding to sucrose (a) and monoesters (b) are indicated. Analytical conditions are described in the Experimental Procedures section. For abbreviations see Figures 1 and 3.

thesis of 2-*O*-acylsucroses using special electrophiles (3-acylthiazolidine-2-thiones) and a strong base (sodium hydride). In our work, in addition to the major components (2-*O*-acylsucroses), we also observed other sucrose monoesters as minor products; the identity of the most abundant was 3-*O*-acylsucrose as determined by ^{1}H NMR (see Experimental Procedures section).

We also observed that the formation of 3-*O*-acylsucroses could not be avoided because they were produced by acyl migration from the 2-*O*-acyl derivatives under reaction conditions (see Fig. 5). These results are in agreement with those from Baczko *et al.* (15), where the same isomerization in the Mitsunobu acylation of sucrose (in the presence of triethylamine) was reported.

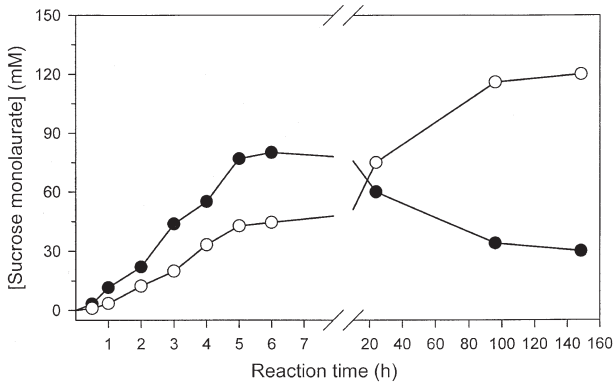

FIG. 5. Variation of the molar ratio between 2-*O*-lauroylsucrose (●) and 3-*O*-lauroylsucrose (○) in the course of the reaction. Conditions: 1.5 M sucrose, 0.15 M vinyl laurate, 40°C, 100 mg/mL Na_2HPO_4.

Acylation of the secondary 2-OH of sucrose makes the glycosidic bond notably more resistant to enzymatic hydrolysis than that of the commercially available 6-*O*-acylsucrose (16). In addition, owing to its extraction efficacy and its compatibility with protein structure and activity, 2-*O*-lauroylsucrose (and its derivatives) proved useful for the extraction of membrane proteins (17).

Comparison of conversion, yield, cost of catalyst, and purity of sucroesters described in this work suggests that this method is a feasible alternative to current industrial sucrose fatty acid ester synthesis, especially when monoester-enriched derivatives of specific functionality are required.

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