

# Influence of Formulation on the Thermal Behavior of Ice Cream Mix and Ice Cream

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**ABSTRACT:** The influence of fat and emulsifier types on particle size and thermal behavior of aged mixes and the corresponding ice creams was investigated. Mixes and ice creams based on partially unsaturated monodiglycerides (MDG) were characterized by an increased percentage of agglomerated fat globules compared with saturated MDG-based systems. DSC thermograms obtained for refined coconut oil in mix showed a displacement of the main crystallization event toward lower crystallization temperatures compared with fat in the bulk phase. This supercooling effect was more or less pronounced for the three other fats used (hydrogenated coconut oil, refined palm oil, and anhydrous milk fat). In emulsified systems, an additional exotherm was observed that was interpreted in terms of MDG crystallization. The fact that this peak appeared at different temperatures ranging from 32 to 41°C as a function of the fat selection suggested that different fat-emulsifier interactions would occur. In the case of ice creams, although the water peak interfered with the fat peak, melting DSC curves allowed the discrimination between the fat types used in the formulation.

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**KEY WORDS:** Fat, ice cream, ice cream mix, lipid emulsifier, thermal behavior.

Ice cream is a complex polyphasic food system in which part of the dispersed phase consists of fat globules in a crystalline state. These globules are organized in a partially coalesced/agglomerated continuous 3-D network formed during the freezing and whipping steps of ice cream processing. This fat globule organization supports other microstructural elements in ice cream, such as air bubbles, and contributes greatly to the quality of the final product. The partial coalescence phenomenon and the extent of fat globule instability are greatly influenced by the amount of crystallized matter (1,2), the process (3), the size and shape of the fat crystals (2,4), the orientation of the crystals at the interface, and the surfactant type and concentration (5–8). Since each type of fat exhibits a specific polymorphism function of its TAG composition, the thermal behavior of fats during ice cream processing should influence the physicochemical properties of the intermediate and final products.

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In previous studies, we showed that the nature of the fat and the degree of unsaturation of the emulsifier led to different properties of the oil-in-water emulsions (9) and ice creams (10). Although particle size characterization was relevant to discriminate between emulsifiers, this method gave poor information regarding the influence of fat type in oil-in-water emulsions and ice creams. In contrast, determination of rheological parameters and melting times appeared to be two convenient methods to illustrate the influence of fat nature although melting times were related only to room temperature. In particular, a high linear correlation was found between the storage modulus measured at 20°C and the melting time (10).

The aim of the present study was to investigate the effects of fat and emulsifier selection on the thermal characteristics of mixes and ice creams. Four types of fat and two monodiglyceride (MDG) mixtures—saturated (SMDG) and partially unsaturated (PUMDG)—were chosen since they are commonly used in ice cream products. Mixes were studied under rapid cooling conditions to mimic the thermal events that occurred during ice cream processing. The corresponding ice creams were analyzed as their temperature increased since ice creams are subjected to warming during their consumption. The mix and ice cream structures were also characterized by particle size analysis to illustrate the partial coalescence and/or agglomeration of fat globules.

## MATERIALS AND METHODS

**Ice cream preparation.** Eight different ice cream mixes were prepared based on fat (8 wt%; hydrogenated coconut oil, refined coconut oil, refined palm oil, or anhydrous milk fat), emulsifier (0.3 wt%; SMDG or PUMDG—60%  $\alpha$ -MG), skim milk powder (10 wt%), sucrose (12 wt%), corn syrup solids (6 wt%; 40 dextrose equivalent, 80 Brix solid content), guar gum and locust bean gum mixture (0.2 wt%). All ingredients, except the fat, were dry blended, mixed with water, and immediately blended at 65°C with the melted fat portion for 15 min. Mixes were homogenized at 70°C, 170/30 bar, and pasteurized at 85°C for 30 s, cooled, and immediately stored at 4°C in a water bath. They were aged for one night at 4°C under stirring. Batches of ice cream mix (20 L) were frozen in a continuous freezer (overrun: 100%, outlet temperature: –5°C). Ice creams were hardened at –40°C and stored at –25°C. For each recipe, at least two different batches were prepared.

**Particle size analysis.** Particle size distribution of aged mixes and ice creams was measured by integrated light scattering using a Mastersizer S (Malvern Instruments, Malvern, United Kingdom). Samples were directly diluted in the sample chamber with water at approximately 1:1000. Water temperature used for dilution was at 13°C. Ultrasonication was used on ice cream samples to ensure the absence of air bubbles. Particle mean diameter (evaluated by the volume weighted average diameter,  $d_{4,3}$ ) and the cumulative percentage of the particles with diameters greater than 2.0  $\mu\text{m}$  (% particles >2  $\mu\text{m}$ ) were determined.

**Thermal analysis.** A TA Instruments differential scanning calorimeter (model MDSC 2920; Guyancourt, France) was used. Mixes ( $\approx 10$  mg) aged at 4°C for 16 h were sealed in aluminum pans. The samples were first rapidly heated to 60°C and then cooled from 60 to  $-20^\circ\text{C}$  at  $5^\circ\text{C}\cdot\text{min}^{-1}$ . Ice creams ( $\approx 80$  mg) stored at  $-20^\circ\text{C}$  were equilibrated at  $-30^\circ\text{C}$  for 10 min in the DSC instrument. The samples were heated from  $-30$  to  $65^\circ\text{C}$  at  $1^\circ\text{C}\cdot\text{min}^{-1}$ . Heat flow was recorded as a function of increasing or decreasing temperature. For each mix and ice cream preparation, the experiment was conducted at least in duplicate. The percentage of the crystallized matter in ice cream mixes was calculated, according to the thermogram of fat in the mix, on the one hand, and the thermogram of the fat in the bulk phase, on the other hand, from the ratio of the areas under the mix peak and the bulk peak. This latter was balanced by the proportion of fat in the formulation. This calculation was based on the hypothesis that no event other than fat crystallization occurred in the temperature range considered. Similar calculation was performed from ice cream melting DSC thermograms.

**Statistical analysis.** ANOVA of all data, using Fisher's LSD procedures, was made on  $d_{4,3}$ , cumulative % particles >2  $\mu\text{m}$ , onset temperatures, and areas under the DSC peaks to discriminate among the eight formulations.

## RESULTS AND DISCUSSION

**Influence of fat and emulsifier selection on mix and ice cream particle size.** The influence of fat nature and emulsifier type on particle size analysis was studied on mixes after aging (16 h at 4°C, under stirring) and on the corresponding ice creams (Table 1). The  $d_{4,3}$  was chosen to follow changes in particle size diameter. The % particles >2  $\mu\text{m}$  was calculated to characterize fat globule agglomeration and/or partial coalescence (11). In the case of SMDG-based mixes, the fat nature did not influence the droplet mean diameters, which were associated with a low % particles >2  $\mu\text{m}$ . With PUMDG, higher  $d_{4,3}$  values were observed. These average values corresponded to a biphasic distribution (Fig. 1) and an increase in the % particles >2  $\mu\text{m}$  (Table 1). Admittedly, emulsifiers do not play a major role in ice cream mix stabilization just after homogenization at high temperatures due to an excess of protein in the mix formulation. Thus, the stabilization of the fat globules against coalescence is achieved thanks to the presence of sufficient protein to cover the droplet surface fully (9,12). The influence of MDG type could account for different protein coverage of the fat droplets in the presence of competitively adsorbing emulsifiers, as observed in emulsified systems (9,12).

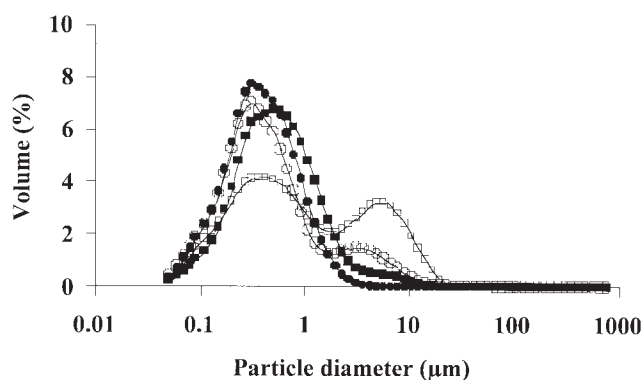
Irrespective of the formulation, the whipping/freezing steps led to an increase in  $d_{4,3}$  in ice creams compared with the corresponding mixes (Table 1, Fig. 1). For a given fat, PUMDG was associated with a greater increase in particle size in ice creams than SMDG, which corresponded to a two- or threefold increase in % particles >2  $\mu\text{m}$ . This suggested that PUMDG in ice cream was more efficient in promoting aggregation and/or partial coalescence of fat droplets than SMDG (11–14). The increase in size between mixes and ice cream was independent of fat type used for SMDG-based products, i.e., close to 64% on average. In contrast, it was influenced by the degree of unsaturation of fat,

**TABLE 1**  
Effect of Fat Type and Emulsifier Nature on Particle Size Characteristics in Aged Mixes and Ice Creams

Fat/emulsifier	Aged mixes		Ice creams	
	$d_{4,3}$ ( $\mu\text{m}$ ) <sup>a</sup>	% particles >2 $\mu\text{m}$ <sup>a,b</sup>	$d_{4,3}$ ( $\mu\text{m}$ ) <sup>a</sup>	% particles >2 $\mu\text{m}$ <sup>a,b</sup>
Refined coconut oil				
SMDG	0.58 $\pm$ 0.01	1.6 $\pm$ 0.1	1.43 $\pm$ 0.02	14.6 $\pm$ 1.3
PUMDG	1.50 $\pm$ 0.23	15.5 $\pm$ 2.6	2.53 $\pm$ 0.49	36.7 $\pm$ 2.1
Hydrogenated coconut oil				
SMDG	0.58 $\pm$ 0.03	1.8 $\pm$ 0.3	1.30 $\pm$ 0.08	14.3 $\pm$ 1.2
PUMDG	1.72 $\pm$ 0.05	11.2 $\pm$ 2.1	2.10 $\pm$ 0.40	24.7 $\pm$ 4.6
Refined palm oil				
SMDG	0.60 $\pm$ 0.00	2.2 $\pm$ 0.4	2.15 $\pm$ 0.40	25.1 $\pm$ 4.6
PUMDG	0.99 $\pm$ 0.07	6.9 $\pm$ 2.0	3.29 $\pm$ 0.57	43.7 $\pm$ 6.6
Anhydrous milk fat				
SMDG	0.58 $\pm$ 0.01	3.6 $\pm$ 1.9	1.60 $\pm$ 0.19	13.6 $\pm$ 1.7
PUMDG	0.96 $\pm$ 0.21	5.8 $\pm$ 1.8	2.80 $\pm$ 0.15	39.1 $\pm$ 2.2

<sup>a</sup>Values are means  $\pm$  SD ( $n = 2$ ).

<sup>b</sup>Percentage of particles with diameters greater than 2  $\mu\text{m}$ . SMDG, saturated monodiglyceride mixture; PUMDG: partially unsaturated monodiglyceride mixture.



**FIG. 1.** Particle size distribution observed after dispersion in distilled water of mixes (closed symbols) and ice creams (open symbols) based on refined coconut oil and saturated monodiglycerides (●, ○) or partially unsaturated monodiglycerides (■, □).

which, in the case of PUMDG, ranged from 20 to 70%. Previous results obtained with vegetable fats suggested that fat globule destabilization was all the more important since the FA chains of the fat were unsaturated and long (10). The results obtained with anhydrous milk fat were in good agreement with this statement. They illustrated that globule stabilization occurred through interactions between the fat and the lipid emulsifier. These interactions would be favored with SMDG, i.e., when the largest hydrophobic parts of the FA chains of emulsifier and fat would fit with each other.

**Mix crystallization.** DSC crystallization curves were obtained on aged mixes and compared with that of fat in bulk phase. The first step was to warm the mix before cooling it. Fat crystallization can be influenced by fat droplet size (8) and

emulsion stability (15). For each formulation, we checked that the particle size in the mix remained unchanged in the conditions of the thermal analysis (results not shown). Thus, it could be assumed that the thermal reactions of fat were not related to a modification of the interfacial composition and/or mix destabilization. Moreover, rapid cooling conditions ( $5^{\circ}\text{C}\cdot\text{min}^{-1}$ ) were used to mimic the thermal events that occurred during ice cream processing.

The amount of crystallized matter in aged mixes was calculated (Table 2). For fats composed mostly of saturated FA (coconut oils), this amount was low, i.e., close to 30%. In contrast, for refined palm oil and anhydrous milk fat, the crystallization level was high, ranging from 50 to 65%. This may be related to different crystallization kinetics between bulk and emulsions (16). Typical crystallization curves obtained for refined coconut oil in bulk phase or in emulsified systems are shown in Figure 2. In the bulk phase, refined coconut oil crystallized in a principal peak (peak 1) composed of two overlapping exothermic peaks. This suggested that some partitioning of TAG species should occur during the crystallization process (17). In emulsified systems, a small crystallization event occurred above  $30^{\circ}\text{C}$  (peak 2) that was not detected in the bulk fat thermogram. Moreover, a displacement of peak 1 toward lower crystallization temperatures was observed for mixes compared with the bulk phase. The presence of an extra thermal event around  $-15^{\circ}\text{C}$  enlarged the crystallization profile of emulsified systems whereas almost all fat in bulk phase was crystallized at  $-10^{\circ}\text{C}$ . The influence of MDG type on the cooling curves was illustrated by different peak 1 profiles and different onset temperatures of peak 2.

Table 2 reports the onset temperatures and enthalpy values determined for peaks 1 and 2 for both bulk and emulsified fats.

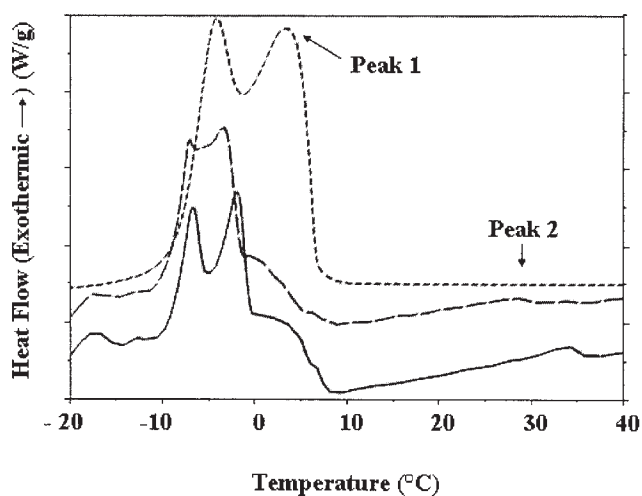
**TABLE 2**  
Effect of Fat Type and Emulsifier Nature on Crystallized Fat, Crystallization Temperatures, and Enthalpies of DSC Cooling Curves for Bulk and Emulsified Fats in Aged Mixes

Fat/physical form		Crystallized matter (%) <sup>a</sup>	Peak 1 <sup>b</sup>			Peak 2 <sup>b</sup>	
			Onset temperature ( $^{\circ}\text{C}$ ) <sup>c</sup>	End of peak ( $^{\circ}\text{C}$ ) <sup>c</sup>	Area ( $\text{J}\cdot\text{g}^{-1}$ ) <sup>c</sup>	Onset temperature ( $^{\circ}\text{C}$ ) <sup>c</sup>	Area ( $\text{J}\cdot\text{g}^{-1}$ ) <sup>c</sup>
Refined coconut oil							
Bulk			$10.0 \pm 1.8$	$-18.5 \pm 1.0$	$97.81 \pm 2.90$	—	—
Emulsified	SMDG	$34 \pm 4$	$7.9 \pm 0.4$	$-14.8 \pm 3.3$	$2.65 \pm 0.39$	$36.1 \pm 0.2$	$0.18 \pm 0.01$
	PUMDG	$31 \pm 1$	$8.9 \pm 0.0$	$-17.6 \pm 2.4$	$2.55 \pm 0.08$	$31.9 \pm 0.8$	$0.20 \pm 0.02$
Hydrogenated coconut oil							
Bulk			$19.3 \pm 1.1$	$-22.2 \pm 1.5$	$103.07 \pm 1.90$	—	—
Emulsified	SMDG	$29 \pm 4$	$16.7 \pm 2.8$	$-14.6 \pm 3.1$	$2.61 \pm 0.55$	$36.3 \pm 0.9$	$0.15 \pm 0.02$
	PUMDG	$37 \pm 1$	$17.0 \pm 1.4$	$-16.0 \pm 0.4$	$2.86 \pm 0.05$	$33.7 \pm 0.1$	$0.05 \pm 0.00$
Refined palm oil							
Bulk			$20.7 \pm 0.0$	$-25.3 \pm 0.0$	$50.62 \pm 3.80$	—	—
Emulsified	SMDG	$57 \pm 6$	$22.6 \pm 0.5$	$-16.0 \pm 1.2$	$2.00 \pm 0.25$	$38.9 \pm 0.3$	$0.14 \pm 0.00$
	PUMDG	$65 \pm 3$	$22.3 \pm 1.7$	$-13.2 \pm 0.1$	$2.62 \pm 0.11$	$36.6 \pm 0.3$	$0.07 \pm 0.02$
Anhydrous milk fat							
Bulk			$18.0 \pm 0.0$	$-26.1 \pm 0.0$	$59.76 \pm 0.70$	—	—
Emulsified	SMDG	$60 \pm 4$	$20.9 \pm 0.6$	$-16.6 \pm 1.1$	$2.57 \pm 0.16$	$41.3 \pm 1.8$	$0.14 \pm 0.01$
	PUMDG	$50 \pm 3$	$16.8 \pm 3.2$	$-18.4 \pm 1.5$	$2.41 \pm 0.16$	$36.6 \pm 1.7$	$0.07 \pm 0.01$

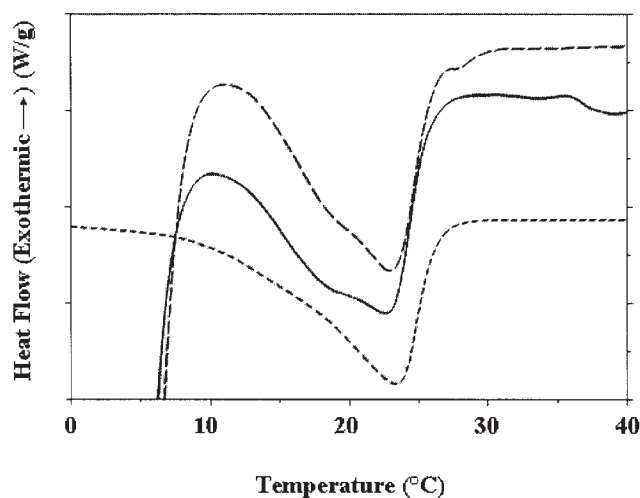
<sup>a</sup>The percentage of the crystallized matter contained in mixes was calculated according to: (area under peak 1 in mix  $\times 100$ )/(area under peak 1 in bulk fat  $\times 0.08$ ). The value 0.08 is derived from the 8% fat content in the formulations. The area under peak 2 in mix was neglected.

<sup>b</sup>Peaks 1 and 2 are defined as in Figure 2.

<sup>c</sup>Values are means  $\pm$  SD ( $n = 2$ ). SMDG, saturated monodiglyceride mixture; PUMDG, partially unsaturated monodiglyceride mixture.



**FIG. 2.** Exothermic peaks obtained by DSC during cooling of refined coconut oil in the bulk phase (---) and in emulsions based on saturated monodiglycerides (—) or partially unsaturated monodiglycerides (— · —).



**FIG. 3.** Endothermic peaks obtained by DSC during melting of refined coconut oil in the bulk phase (---) and in ice cream based on saturated monodiglycerides (—) or partially unsaturated monodiglycerides (— · —).

For fats principally composed of saturated FA, such as refined and hydrogenated coconut oils, a displacement of the onset temperature of peak 1 toward lower crystallization values was observed in mixes. This agrees with crystallization of emulsified oil requiring a degree of supercooling (2,16). The delayed fat crystallization occurring in mix systems compared with the bulk fat sample could be explained by homogeneous nucleation through spontaneous formation of nuclei in the case of emulsions, whereas in bulk oil systems nucleation is predominantly heterogeneous (16,18,19). A noncatalytic action of adsorbed molecular species also has been proposed (14). In the case of refined palm oil and anhydrous milk fat, supercooling in mix samples was not observed probably due to the larger range of crystallization temperatures of these fats. The additional exotherm (peak 2), found in all emulsion systems, could be attributed mainly to MDG crystallization. Indeed, MDG crystallize at higher temperatures than the fats used in this work. The onset temperature of peak 2 was higher in SMDG-based systems than in PUMDG ones. This agrees with saturated FA that begin to crystallize at higher temperatures than unsaturated FA. However, since different onset temperature values and enthalpy values were recorded as a function of the fat used, specific interactions between the emulsifier and the fat could not be excluded. This was especially the case for mix where PUMDG was associated with refined coconut oil (Table 2).

**Ice cream fusion.** DSC was used to characterize the thermal behavior of ice cream upon heating. Regardless of ice cream formulation, DSC patterns exhibited a peak ranging from  $-30$  to  $10^{\circ}\text{C}$  that was attributed, to a large extent, to free water (20). When compared with the fusion thermograms of bulk fats, i.e., neglecting the emulsifying effects, the part of melted fat overlapped by the broad water melting peak in ice cream could be estimated at 11% for both refined and hydrogenated coconut oils, 38% for anhydrous milk fat, and 60% for refined palm oil. The greater overlapping obtained for the anhydrous milk fat

and refined palm oil was due to their large temperature melting range (17,21). Thus, DSC thermograms were analyzed only for the two types of coconut oil in the temperature range between 10 and  $40^{\circ}\text{C}$ . Typical melting profiles are presented in Figure 3 for refined coconut oil-based ice creams formulated with SMDG or PUMDG. Melting of refined coconut oil bulk phase is also reported. The profiles of fat melting in ice cream and bulk system were similar although a shoulder in the main peak and a small thermal event around  $30^{\circ}\text{C}$  were detected in ice cream curves. In particular, nearly identical maximum melting temperatures ( $\approx 23^{\circ}\text{C}$ ) were found for bulk and emulsified systems, regardless of emulsifier nature. The difference in curve shapes between fat in bulk phase and in ice cream samples could reveal some TAG partitioning in ice cream and/or a more complex TAG polymorphism in emulsified system. When compared with the crystallization curves of mixes based on refined coconut oil (Fig. 2), the maximum melting temperatures of the main thermal event were shifted positively. This indicated that, starting from melted fat, a supercooling is needed to initiate crystallization (14).

Although it is well accepted that the type of fat phase influences ice cream characteristics, very few data actually demonstrate this effect. The eight different recipes herein highlighted the effect of fat and emulsifier selection on mix and ice cream properties. Particle size determination was relevant to discriminate between emulsifiers but gave only poor information regarding the influence of fat type in ice cream products. On the other hand, DSC profiles of emulsified fat in the mix allowed discrimination between the formulations. Crystallization patterns of mix could reveal the existence of different interactions between fat FA and emulsifier specific to each fat-emulsifier couple selected. Concerning ice creams, their melting behavior was also characteristic of the fat used in the formulation. After hardening at  $-40^{\circ}\text{C}$  and storage at  $-20^{\circ}\text{C}$ , the fat globules in ice cream regained the thermal properties of the bulk fat. Nevertheless,

although the differences observed between the emulsified fat and bulk fat melting profiles were quite small, they suggested that the freezing/whipping steps could induce some differences in fat organization.

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