



## Research paper

# The effect of certified pasture-fed cream on the physical properties of mix and ice cream during storage

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## ABSTRACT

Despite the growing popularity of pasture-fed dairy, its effects on ice cream properties remain underexplored. This study investigated the physical properties of emulsions and ice creams made with certified pasture-fed cream versus conventional cream. Creams were characterised by fatty acid profiles and thermal properties. All ice cream formulations were standardised to 10.5 % milk fat, 11 % milk solids-not-fat, 13 % sucrose, 0.2 % stabilisers and 0.2 % emulsifiers. Pasture-fed cream showed lower saturated and higher unsaturated and branched-chain fatty acids, leading to lower solid fat content, crystallization enthalpy, and melting temperature. The aged pasture-fed mix exhibited lower solid fat, viscosity, and viscoelastic moduli. Ice creams made with pasture-fed cream showed lower hardness, particularly after one month, and a higher melting rate. Over 12 weeks, hardness and fat agglomeration increased in all samples. These findings may support the development of ice creams formulated with pasture-fed dairy ingredients, contributing to product innovation and differentiation in the dairy sector.

## 1. Introduction

Ice cream is a popular frozen dairy-based dessert consumed worldwide by people of all ages. Milk fat, usually supplied as cream, is one of the main ingredients of ice cream, contributing approximately 10–18 % of the formulation, and some jurisdictions provide for a minimum amount of fat depending on the ice cream product category (e.g., 15 % for premium and 18 % for super-premium ice cream) (H. D. Goff & R. W. Hartel, 2013). Milk fat is a key food component and energy source for humans, significantly influencing the sensory properties, structure, flavour release, and stability of ice cream (Akbari et al., 2019; Genovese et al., 2022). It is also the primary source of vaccenic acid (VA) and conjugated linoleic acid (CLA) isomers, which may help prevent chronic inflammatory diseases. Pasture feeding improves milk fat composition by reducing saturated fatty acids (FAs), particularly medium- and long-chain FAs such as lauric, palmitic, and stearic acids, thus enhancing lipid nutritional indices (Balivo et al., 2023).

Consumers are increasingly expressing a preference and a willingness to pay a premium for certified pasture-fed dairy products, as they appreciate that well-managed pasture-feeding systems are more

environmentally friendly and promote better animal welfare (Infascelli et al., 2021; Joubran et al., 2021). Certification schemes aim to regulate feeding practices and ensure the inclusion of fresh forages in the diet. For example, the Irish and American Grass-Fed Standards require at least 90 % grass forage (fresh weight), while Italy's Metodo Nobile® and the EU's STG Hay Milk mandate pasture-based feeding for at least 70–75 % of dry matter intake (Alothman et al., 2019; Balivo et al., 2023; Benbrook et al., 2018). In a recent study conducted on Irish (n = 345) and American (n = 432) consumers, McGuinness et al. (2022) found that the 'Grass-Fed Standard' attribute on the label increased the perceived healthiness and naturalness of cheddar cheese. These findings suggest that certified pasture-fed dairy ingredients may offer a promising strategy for developing competitive and appealing products such as ice cream. However, their higher content of unsaturated fatty acids could affect the physical properties of the final product.

There are few studies in the ice cream literature that have investigated the effect of using milk fat with modified fatty acids due to the animal's diet. Some studies have increased the unsaturated fatty acid content of milk fat through oilseed supplementation in the cows' diet, and subsequently produced ice creams that exhibited a higher melting

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rate, lower solid fat content, and reduced viscosity (Gonzalez et al., 2003; Smet et al., 2010; Vargas-Bello-Pérez et al., 2019). Other studies have used the olein and stearin fractions of milk fat as a method to produce ice creams or simulated emulsions with a different fatty acid profile (Bazmi & Relkin, 2006; Marín-Suárez et al., 2016; Nadeem, Situ, & Abdullah, 2015). According to these studies, milk fat with a higher content of unsaturated fatty acids was found to have a lower solid fat content, which in turn led to reduced mix viscosity, lower hardness, and a faster melting rate in ice cream. To date, no studies have investigated the use of certified pasture-fed cream derived from cows extensively grazed on biodiverse pastures in the production and physical stability of ice cream. This study addresses this gap by formulating ice creams with certified pasture-fed cream and comparing their physical properties to those made with conventional, non-certified cream. The aim is to provide new insights into the functional role of pasture-fed dairy ingredients in ice cream formulation, supporting innovation and product differentiation within the dairy sector.

## 2. Materials and methods

### 2.1. Materials

The cream (35 % fat), certified Metodo Nobile®, was obtained from a producer in Lazio, Italy, who is a member of the Metodo Nobile consortium (<https://lattenobile.it/panna-nobile/>). This product is not sold on a large scale but is available on order. The certification ensures that cows are fed a diet consisting of at least 70 % of dry matter intake from fresh forages, with the forages being composed of diverse pasture containing at least five major plant species. The control creams were purchased from local supermarkets in Italy and Belgium. Furthermore, a cream from a local farm (Ferme Notre-Dame, Ligny, Belgium) was obtained, where cows are fed indoors with a forage-to-concentrate ratio of 80:20. The forage, cultivated on-site, is provided as a mix of silage and fresh forage.

The abbreviations used for the cream samples and their derived products (mixes and ice creams) are reported in Table 1.

Skimmed milk powder (Régilait) and sucrose were purchased from local supermarkets. Emulsifiers (mono- and diglycerides of fatty acids) and stabilisers (mixture of locust bean gum, guar gum, sodium alginate, agar agar) were purchased respectively from Gioia Group s.r.l. (Torino, Italy) and Special Ingredients (Savona, Italy). Creams from the same lot were purchased fresh and stored at  $-20\text{ }^{\circ}\text{C}$  until use. Prior to each production run, the cream was thawed and used immediately, without repeated freeze–thaw cycles. Ice cream mixes and final products were prepared in three independent batches over three consecutive weeks.

Ammonia solution 25 %, cyclohexane, petroleum ether, sodium sulphate anhydrous, methanol and hydrochloric acid were obtained from Sigma-Aldrich (Merck KGaA, Darmstadt, Germany). Ethanol 96 % and diethyl ether free from peroxides were supplied by VWR chemicals (Radnor, PA, USA). All reagents were of analytical grade.

### 2.2. Ice cream formulation and production

All ice cream formulations were prepared to a composition of 10.5 % milk fat and 11 % milk solids-not-fat (from skimmed milk powder and

the non-fat portion of cream). The mixes differed in the type of cream used as the source of milk fat in the ice cream.

The ice creams consisted of 46.6 % water, 30 % cream (35 % fat), 13 % sucrose, 10 % skimmed milk powder, 0.2 % emulsifiers and 0.2 % stabilisers. The ingredients were mixed at  $65\text{ }^{\circ}\text{C}$  for 15 min on a stirred heating plate. Then, the ice cream mixes were homogenised ( $60\text{ }^{\circ}\text{C}$ ) at 200 MPa (M110EH Pilot Scale Homogenizer, Germany), cooled and aged at  $4\text{ }^{\circ}\text{C}$  for 24 h. After the ageing, the ice cream mix emulsion was processed in a small-scale batch freezer (SOLIS Gelateria Pro type 850, Zurich, Switzerland) equipped with a built-in compressor. Approximately 1 kg of mix was frozen per batch. The ice cream machine gradually decreased the temperature to  $-23\text{ }^{\circ}\text{C}$  over a 50 min cycle, until the draw temperature (desired ice cream consistency) was reached. Hardening was completed in the same machine for an additional 50 min at  $-35\text{ }^{\circ}\text{C}$ .

The ice creams produced were packaged into 1000 mL plastic containers and stored in a freezer at  $-18\text{ }^{\circ}\text{C}$  before analyses. Mixes and ice creams were produced in three independent production runs, carried out on three consecutive weeks, each using freshly thawed cream batches. This approach ensured independent replicates and avoided repeated freeze–thaw cycles of the same cream lot. Ice creams were analysed at different storage times.

### 2.3. Analysis of cream fat extracts

#### 2.3.1. Fatty acid composition

The milk fat was extracted from the cream according to the reference method (ISO 2450, 2008). A 25 % ammonia solution was added to the cream sample and mixed. Then, 96 % ethanol was added and mixed, followed by the addition of diethyl ether and mixing. Petroleum ether was added, mixed, and the mixture was centrifuged at 600 rpm for 5 min to recover the supernatant. The extraction was repeated two more times on the remaining lower phase. The organic extract was pooled in a boiling flask and evaporated under reduced pressure in a rotary evaporator (Buchi Rotavapor R-210 coupled to Buchi Vacuum pump V-700, Switzerland).

For the preparation of fatty acid methyl esters (FAME) and GC analysis, the microwave-assisted extraction and derivatization (MAED) method as described in Fina et al. (2022) was followed.

Briefly, 0.5 g of the sample was weighed into SR-12 eT TFM vessels, and 10 mL of acidic methanol solution and 25 mL of cyclohexane were added. The vessels were sealed and placed in a microwave-assisted extraction system (ETHOS X with SR-12 eT TFM rotor, Milestone Srl, Bergamo, Italy) for the microwave-assisted extraction and derivatization (MAED) of fatty acids. Samples were heated to  $120\text{ }^{\circ}\text{C}$  with a ramp-up of 2 min and held at this temperature for 15 min. The vessels were then cooled on ice for 15 min to minimize losses of volatile FAMES (e.g., C4, C6) before centrifugation for 5 min to separate the phases. The upper phase containing FAME was analysed using a comprehensive two-dimensional gas chromatographic system (GC  $\times$  GC) (Nexis GC-2030, Shimadzu, Kyoto, Japan) equipped with an AOC-30i autosampler (Shimadzu, Kyoto, Japan), coupled to FID. The system included an INSIGHT reverse fill-flush flow modulator (SepSolve Analytical Ltd, UK). The GC  $\times$  GC columns configuration consisted of a SepSolve  $^1\text{D}$ -FAMES polar column (20 m  $\times$  0.18 mm  $\times$  0.1  $\mu\text{m}$ ) and a SepSolve  $^2\text{D}$ -FAMES non-polar column (5 m  $\times$  0.25 mm  $\times$  0.1  $\mu\text{m}$ ) (SepSolve Analytical Ltd, UK). The bleedline was 4.20 m  $\times$  0.1 mm uncoated capillary segment. The flow rates were as follows: first dimension flow was set at 0.5 mL/min; the auxiliary flow controller generating the second-dimension flow was set at 20 mL/min. Helium was used as carrier gas. The modulation period was set at 3 s, including 100 ms of reinjection time. The oven temperature program started at  $40\text{ }^{\circ}\text{C}$  for 3 min, ramped to  $260\text{ }^{\circ}\text{C}$  at  $9\text{ }^{\circ}\text{C}/\text{min}$ , and held for 2 min. The injection was performed in split mode (1:10 ratio), injecting 0.5  $\mu\text{L}$  at  $250\text{ }^{\circ}\text{C}$ . Detection was performed using the FID set at  $270\text{ }^{\circ}\text{C}$ . Data acquisition frequency was set to 100 Hz.

Data was acquired by LabSolution Version 5.111 and processed by

**Table 1**

Abbreviations used for cream samples and their derived products (mixes and ice creams).

Sample abbreviation	Description
Ni	Certified pasture-fed cream ( $\geq 70\%$ dry matter intake from outdoor biodiverse fresh pasture)
Nb	Belgian farm cream (indoor-fed, $\sim 80\%$ forage as silage/fresh)
Ci	Italian commercial cream
Cb	Belgian commercial cream

ChromSpace Version 1.5.1 by Markes International Limited.

Fatty acid peaks in chromatograms were identified using the Supelco 37 Component FAME MIX (Supelco, Bellefonte, PA) and according to their position in the 2D space. Standards for CLA (C18:2 cis-9-trans-11) and trans vaccenic acid (C18:1 trans-11) were obtained from NuChek Prep (Elysian, MN). Fatty acids (FA) were expressed as a percentage of total methylated fatty acids (g/100g FAs). The sum of saturated (SFA), monounsaturated (MUFA), and polyunsaturated (PUFA) FAs was calculated. The atherogenic and spreadability index were calculated as reported by Couvreur et al. (2006).

### 2.3.2. Thermal properties

The thermal properties of the cream fat extract samples were investigated by differential scanning calorimetry (DSC) using a Q1000 DSC (TA Instruments, New Castle, USA). Calibration was made with indium (m.p. 156.6 °C,  $\Delta H_f = 28.45 \text{ J g}^{-1}$ ) and n-dodecane (m.p. -9.65 °C,  $\Delta H_f = 216.73 \text{ J g}^{-1}$ ) standards. Nitrogen was used as a purge gas to prevent condensation in the cells. Prior to DSC analysis, the sample was heated at 60 °C for 5 min to melt all crystals and nuclei. An empty hermetically sealed Tzero aluminum pan was used as a reference. Each sample (6–8 mg) was placed in a pre-weighed Tzero aluminum pan and hermetically sealed using a Quick Press pan crimper (Tzero, TA Instruments, New Castle, USA). The samples were heated at 60 °C for 5 min, cooled at 2 °C/min from 60 to -40 °C, and then heated at 2 °C/min from -40 to 60 °C (Couvreur et al., 2006). A slow cooling and heating rate was chosen to achieve better separation and resolution of the peaks of the milk fat fractions (Tomaszewska-Gras, 2013).

The temperature of the beginning of crystallization ( $T_{con}$ ) and of each peak ( $T_{c1}$ ,  $T_{c2}$ ), as well as the enthalpy of crystallization ( $\Delta H_C$ ,  $\text{J g}^{-1}$ ), were determined from cooling curves. The onset temperature ( $T_{Mon}$ ), the melting point for the first peak ( $T_{M1}$ ) of the low melting fraction, the temperature of the second melting peak ( $T_{M2}$ ) of the medium melting fraction, and the final melting temperature at the end of the high melting fraction ( $T_{Mend}$ ), and enthalpy ( $\Delta H_M$ ,  $\text{J g}^{-1}$ ) were determined from the heating curves for the melting process (Advantage Software version 2, TA Instrument). Three replicates for each sample were performed.

### 2.3.3. Solid fat content

The solid fat content (SFC) of the cream fat extract samples was determined based on the AOCS official method Cd 16b-93 (AOCS, 1998) by means of pulsed nuclear magnetic resonance (p-NMR) using a 20-MHz Bruker Minispec mq20 (Bruker, Germany). Daily check and automatic calibration were made using a set of three standards with a SFC of 0.0, 31.1 and 74.8 %, respectively. The NMR tubes were filled with sample up to a height of 4 cm. Samples were heated for 15 min at 100 °C, kept at 60 °C for 5 min, and stabilised at 0 °C for 1 h using a high-precision dry bath (LAIX Technologies UG, Simmerath, Germany). The determinations were carried out in series, at temperatures of 5, 10, 15, 20, 25, 30, 35, 40 and 45 °C. For each cream type, fat was extracted in triplicate from a pooled sample, and the measurements were performed ( $n = 3$ ).

## 2.4. Analysis of mixes and ice creams

### 2.4.1. Thermal properties of mix

The thermal properties of mix samples were investigated using a Q1000 DSC (TA Instruments, New Castle, USA). An empty hermetically sealed Tzero aluminum pan was used as a reference. Ice cream mixes were weighed (15–30 mg) and sealed into Tzero aluminum pans. The samples were heated to 80 °C, equilibrated at that temperature for 5 min, cooled to -60 °C at the rate of 2 °C/min, equilibrated at -60 °C, and heated to 80 °C at a rate of 2 °C/min (Zulim Botega et al., 2013). Thermograms were analysed with TA Universal Analysis Software (Advantage Software version 2, TA Instrument). From the cooling curves, the onset ( $T_{con}$ ), peak crystallization temperature ( $T_{cmax}$ ), and

crystallization enthalpy ( $\Delta H_C$ ,  $\text{J g}^{-1}$ ) were determined. From the heating curves, the onset of melting ( $T_{Mon}$ ), peak melting temperature ( $T_{Mmax}$ ), final melting temperature ( $T_{Mend}$ ), and melting enthalpy ( $\Delta H_M$ ,  $\text{J g}^{-1}$ ) were recorded.

### 2.4.2. Solidified fat in mix

Solidified fat content in the ice cream mixes was determined in triplicate using pNMR spectroscopy (Minispec mq20, Bruker, Germany). Following the homogenization of mix samples, NMR tubes were filled with the samples and kept at 4 °C for 0, 1, 2, 3, 4, 5, 6, 7, and 24 h in a high-precision dry bath (LAIX Technologies UG, Simmerath, Germany). Measurements were taken at these intervals to study the crystallization kinetics of the fat during the ageing phase of the mix at 4 °C (Abd El-Rahman et al., 1997). Three replicates were performed on the three batches produced in the three consecutive weeks.

### 2.4.3. Rheological properties of mix

Following the 24-h ageing period, samples of ice cream mix were analysed at 4 °C for viscosity and viscoelastic properties using an Anton Paar Physica MCR 302 rheometer (Anton Paar GmbH, Graz, Austria) with crosshatched parallel plates (PP25/P2, diameter 25 mm) and a gap of 1 mm. The rheometer was equipped with a Peltier system and water bath (Julabo, Seelbach, Germany) for temperature control. The roughened geometry was chosen to prevent sample slip (Nickerson & Kornfield, 2005).

Rotational tests (shear rate sweeps) were carried out in the shear rate range 0.1–100  $\text{s}^{-1}$ . Apparent viscosity, consistency coefficients and flow behaviour indices were determined using the software of Rheoplus/32 v3.21 (Anton Paar GmbH, Graz, Austria). Analysis of the curves was performed with curve fitting according to the Herschel-Bulkley model:  $\tau = \tau_0 + K \cdot \dot{\gamma}^n$ ; where  $\tau$  = shear stress,  $\tau_0$  = yield stress,  $K$  = viscosity (consistency) coefficient,  $\dot{\gamma}$  = shear rate, and  $n$  = flow behaviour index ( $R^2 = 0.99$ ). Apparent viscosity was calculated from the model at shear rates of 30  $\text{s}^{-1}$  and 50  $\text{s}^{-1}$ , as these rates are reported to closely approximate the shear rate experienced in the oral cavity while eating low viscosity foods similar to ice cream (Amador et al., 2017; Goff et al., 1995).

For oscillatory measurements, a strain sweep (0.1–100 %) at 1 Hz was conducted to determine the linear viscoelastic region (LVR) of the samples. A frequency sweep (0.1–10 Hz) was then performed at a fixed strain of 0.5 %, within the LVR. The viscoelastic parameters, i.e. storage modulus ( $G'$ ), loss modulus ( $G''$ ), and loss tangent ( $\tan \delta = G''/G'$ ), were determined as functions of frequency (Goff et al., 1995).

### 2.4.4. Overrun, first dripping, complete melting time and melting rate of ice cream

The overrun (%), i.e. the incorporation of air into the ice cream, was calculated per sample by comparing the mass of ice cream mix and ice cream in a fixed volume container (250 mL) using the following equation (Akin et al., 2007):

$$\text{Overrun (\%)} = \frac{\text{Mass of unit mix} - \text{Mass of an equal volume of ice cream}}{\text{Mass of an equal volume of ice cream}} \times 100$$

The ice cream (spherical shape, -18 °C, 30 g) was sampled using an ice cream scoop and transferred on a wire mesh screen placed over a funnel to melt it at a temperature of  $21.5 \pm 0.5$  °C. The first dripping time and the complete melting time were determined in seconds (Guler-Akin et al., 2021). The weight of the melted ice cream was recorded every minute to generate a sigmoidal curve representing the kinetics of the melting process. The data points from the linear portion of the curve were fitted using linear regression, and the slope of this line was used to determine the melting rate (g/min) (Balthazar et al., 2018). Analyses were carried out in triplicate.

#### 2.4.5. Fat particle size distribution analysis and fat destabilization in ice cream

Particle size distributions of milk fat globules and agglomerated fat in the mixes and ice creams were determined by laser light scattering using a PSA 1190 particle size analyser (Anton Paar, Graz, Austria). Mixes were analysed immediately after cooling to 4 °C, while the hardened ice cream samples were tempered at 4 °C before analysis (Warren & Hartel, 2018). Deionised water was used as the dispersant, and measurements were performed within an obscuration range of 13–15 %, corresponding to approximately four drops of sample. The refractive indices of milk fat (1.460) and water (1.330) were used for particle and dispersant, respectively.

Mean size volume ( $d_{4,3}$ ,  $\mu\text{m}$ ) and mean size surface ( $d_{3,2}$ ,  $\mu\text{m}$ ) were recorded. The volume fraction of coalesced fat (fat destabilization) in the ice cream samples was calculated as the percent of the distribution larger than the 90th percentile ( $d_{v,(0,9)}$ ,  $\mu\text{m}$ ) of the original mix distribution (Sung & Goff, 2010).

#### 2.4.6. Texture analysis of ice cream

The hardness of ice cream was evaluated using an SMS TA.XT2i texture analyser equipped with a 36 mm Perspex cylinder probe (P/36R) (Stable Micro Systems, Godalming, Surrey UK) under the following conditions: compression power mode test; load cell, 50 kg; trigger force, 5.0 g; pre-test speed, 2.0 mm/s; test and post-test speed, 1.0 mm/s; penetration distance, 10.0 mm (Balthazar et al., 2018). Approximately 35 g of ice cream, stored at  $-18$  °C, was shaped into a uniform block (5 cm diameter  $\times$  30 mm height) with a flat surface inside a cylindrical paper cup. Hardness tests were performed at 20 °C by positioning the indenter above the sample's center, and five replicates were performed. Hardness was calculated as the peak compression positive force (peak max) (N) during probe penetration using Texture Expert 1.22 software (Stable Micro Systems).

#### 2.4.7. Colour analysis of mix and ice cream

The colour properties of mixes and ice creams, in  $L^*$ ,  $a^*$  and  $b^*$  values of CIELAB colour space, were measured using an instrumental reflectance colourimeter (ColorFlex EZ 45/0-LAV, Hunter Associates Laboratory Inc., Virginia, USA), where  $L^*$  represents lightness,  $a^*$  ranges from green (–) to red (+), and  $b^*$  ranges from blue (–) to yellow (+). The black and white reference plates were used to calibrate the instrument before the measurements. The sample was homogenised and poured into the optically clear glass cup covered with a black rubber ring on the side and a disk on top (Hunter Associates Laboratory Inc., Virginia, USA). Five replicates were performed at different points (centre and edges) for each sample. The whiteness index (WI) was calculated using the following equation (Balthazar et al., 2018):

$$WI = 100 - \left[ (100 - L^*)^2 + (a^*)^2 + (b^*)^2 \right]^{\frac{1}{2}}$$

### 2.5. Statistical analysis

Results were expressed as the mean  $\pm$  standard deviation. Significant differences were assessed by analysis of variance (ANOVA) with Tukey's HSD test, for a significance level set at  $p \leq 0.05$ . Statistical analysis and visualization were carried out in XLStat environment (Version, 2019 v.2.2), an add-in software package for Microsoft Excel (Addinsoft Corp., Paris, France).

## 3. Results and discussion

### 3.1. Fatty acids and thermal properties of cream

Fatty acid profiles of the cream samples are shown in Table 2. The main FAs, in decreasing order, were palmitic (16:0), oleic (18:1 c9), stearic (18:0), capric (10:0) and myristic (14:0) acids, comprising

**Table 2**

Fatty acid composition (% weight of total methyl esters) of cream samples used in the production of ice creams.

Fatty acid*	Ni	Cl	Nb	Cb
<b>Saturated fatty acids (SFA)</b>				
C4:0 (butyric)	3.43 $\pm$ 0.08 a	3.83 $\pm$ 0.05 a	3.91 $\pm$ 0.44 a	3.24 $\pm$ 0.20 a
C6:0 (caproic)	2.32 $\pm$ 0.20 a	2.24 $\pm$ 0.03 a	2.14 $\pm$ 0.02 a	2.30 $\pm$ 0.23 a
C8:0 (caprylic)	1.25 $\pm$ 0.11 a	1.27 $\pm$ 0.11 a	1.28 $\pm$ 0.28 a	1.31 $\pm$ 0.01 a
C10:0 (capric)	2.77 $\pm$ 0.06 ab	2.20 $\pm$ 0.15 c	2.26 $\pm$ 0.21 bc	3.03 $\pm$ 0.01 a
C11:0 (undecylic)	0.03 $\pm$ 0.00 c	0.04 $\pm$ 0.00 bc	0.05 $\pm$ 0.00 ab	0.06 $\pm$ 0.01 a
C12:0 (lauric)	3.00 $\pm$ 0.34 a	2.93 $\pm$ 0.31 a	3.00 $\pm$ 0.07 a	3.16 $\pm$ 0.15 a
C13:0 (tridecylic)	0.09 $\pm$ 0.01 b	0.08 $\pm$ 0.00 b	0.09 $\pm$ 0.00 b	0.11 $\pm$ 0.00 a
C14:0 (myristic)	10.15 $\pm$ 0.15 a	9.64 $\pm$ 0.37 a	10.55 $\pm$ 0.43 a	10.07 $\pm$ 0.16 a
C15:0 (pentadecylic)	1.14 $\pm$ 0.01 a	1.08 $\pm$ 0.21 a	1.02 $\pm$ 0.03 a	1.08 $\pm$ 0.06 a
C16:0 (palmitic)	29.99 $\pm$ 0.62 b	34.19 $\pm$ 0.34 a	31.67 $\pm$ 0.72 ab	33.66 $\pm$ 1.69 ab
C17:0 (margaric)	0.58 $\pm$ 0.01 a	0.44 $\pm$ 0.02 b	0.43 $\pm$ 0.02 b	0.47 $\pm$ 0.03 b
C18:0 (stearic)	10.28 $\pm$ 0.04 b	13.74 $\pm$ 0.31 a	13.87 $\pm$ 0.93 a	11.70 $\pm$ 1.10 ab
C20:0 (arachidic)	0.15 $\pm$ 0.03 a	0.12 $\pm$ 0.00 a	0.14 $\pm$ 0.01 a	0.11 $\pm$ 0.01 a
C22:0 (behenic)	0.08 $\pm$ 0.01 a	0.04 $\pm$ 0.00 b	0.07 $\pm$ 0.00 a	0.04 $\pm$ 0.00 b
C24:0 (lignoceric)	0.03 $\pm$ 0.00 a	0.03 $\pm$ 0.00 a	0.04 $\pm$ 0.00 a	0.01 $\pm$ 0.00 a
<b><math>\Sigma</math> SFA</b>	<b>65.27 <math>\pm</math> 0.30 b</b>	<b>71.87 <math>\pm</math> 0.46 a</b>	<b>70.51 <math>\pm</math> 0.87 a</b>	<b>70.36 <math>\pm</math> 0.26 a</b>
<b>Branched-chain fatty acids (BCFA)</b>				
C13:0 iso	0.04 $\pm$ 0.00 a	0.02 $\pm$ 0.00 b	0.02 $\pm$ 0.00 b	0.02 $\pm$ 0.00 b
C14:0 iso	0.16 $\pm$ 0.01 a	0.06 $\pm$ 0.00 b	0.06 $\pm$ 0.00 b	0.06 $\pm$ 0.00 b
C15:0 iso	0.27 $\pm$ 0.01 a	0.17 $\pm$ 0.01 b	0.15 $\pm$ 0.00 b	0.15 $\pm$ 0.00 b
C15:0 anteiso	0.58 $\pm$ 0.02 a	0.37 $\pm$ 0.02 b	0.31 $\pm$ 0.01 b	0.36 $\pm$ 0.01 b
C16:0 iso	0.38 $\pm$ 0.00 a	0.17 $\pm$ 0.00 bc	0.15 $\pm$ 0.01 c	0.18 $\pm$ 0.01 b
C17:0 iso	0.58 $\pm$ 0.08 a	0.32 $\pm$ 0.05 b	0.34 $\pm$ 0.00 b	0.28 $\pm$ 0.02 b
C17:0 anteiso	0.37 $\pm$ 0.06 ab	0.30 $\pm$ 0.06 ab	0.19 $\pm$ 0.03 b	0.39 $\pm$ 0.03 a
C18:0 iso	0.18 $\pm$ 0.03 a	0.09 $\pm$ 0.00 bc	0.06 $\pm$ 0.01 c	0.14 $\pm$ 0.00 ab
<b><math>\Sigma</math> BCFA</b>	<b>2.54 <math>\pm</math> 0.16 a</b>	<b>1.50 <math>\pm</math> 0.04 b</b>	<b>1.28 <math>\pm</math> 0.03 b</b>	<b>1.59 <math>\pm</math> 0.07 b</b>
<b>Monounsaturated fatty acids (MUFA)</b>				
C10:1 9c (caprolic)	0.23 $\pm$ 0.02 a	0.18 $\pm$ 0.00 b	0.20 $\pm$ 0.01 ab	0.19 $\pm$ 0.02 ab
C14:1 9c (myristoleic)	1.08 $\pm$ 0.05 a	0.98 $\pm$ 0.04 ab	0.87 $\pm$ 0.01 b	0.98 $\pm$ 0.01 ab
C16:1 9c (palmitoleic)	1.74 $\pm$ 0.09 ab	1.83 $\pm$ 0.08 ab	1.55 $\pm$ 0.06 b	2.01 $\pm$ 0.12 a
C17:1 10c (cis-10-heptadecenoic)	0.43 $\pm$ 0.02 a	0.43 $\pm$ 0.01 a	0.34 $\pm$ 0.00 b	0.43 $\pm$ 0.03 ab
C18:1 9t (elaidic)	0.56 $\pm$ 0.09 a	0.21 $\pm$ 0.04 b	0.23 $\pm$ 0.04 b	0.32 $\pm$ 0.06 b
C18:1 11t (vaccenic)	0.97 $\pm$ 0.06 b	0.47 $\pm$ 0.08 c	1.25 $\pm$ 0.06 a	0.67 $\pm$ 0.06 c
C18:1 9c (oleic)	23.61 $\pm$ 0.26 a	19.77 $\pm$ 0.25 b	20.37 $\pm$ 0.75 b	20.66 $\pm$ 0.50 b
<b><math>\Sigma</math> MUFA</b>	<b>28.62 <math>\pm</math> 0.29 a</b>	<b>23.87 <math>\pm</math> 0.41 b</b>	<b>24.81 <math>\pm</math> 0.79 ab</b>	<b>25.26 <math>\pm</math> 0.37 b</b>
<b>Polyunsaturated fatty acids (PUFA)</b>				
C18:2 9c,12c (linoleic)	2.14 $\pm$ 0.10 a	1.79 $\pm$ 0.09 a	1.81 $\pm$ 0.12 a	1.73 $\pm$ 0.09 a

(continued on next page)

Table 2 (continued)

Fatty acid*	Ni	Ci	Nb	Cb
c18:3 6c,9c,12c ( $\gamma$ -linolenic)	0.06 $\pm$ 0.00 ab	0.03 $\pm$ 0.00 c	0.07 $\pm$ 0.01 a	0.04 $\pm$ 0.00 bc
C18:3 9c,12c,15c ( $\alpha$ -linolenic)	0.50 $\pm$ 0.07 ab	0.34 $\pm$ 0.01 b	0.65 $\pm$ 0.02 a	0.41 $\pm$ 0.03 b
C18:2 CLA 9c,11t (conjugated linoleic)	0.75 $\pm$ 0.01 a	0.53 $\pm$ 0.08 b	0.81 $\pm$ 0.13 a	0.49 $\pm$ 0.08 b
C20:3 8c,11c,14c (dihomo- $\gamma$ -linolenic)	0.11 $\pm$ 0.01 a	0.08 $\pm$ 0.01 ab	0.07 $\pm$ 0.00 b	0.11 $\pm$ 0.01 a
$\Sigma$ PUFA	3.56 $\pm$ 0.17 a	2.77 $\pm$ 0.01 b	3.40 $\pm$ 0.26 ab	2.79 $\pm$ 0.04 b
Atherogenic index	2.38	2.85	2.72	2.75
Spreadability index	1.31	1.73	1.55	1.63

Data are shown as mean  $\pm$  standard deviation (n = 3). Different letters indicate statistically differences between the samples (p < 0.05). \* = g/100 g of total fatty acids.

Atherogenic index = [C12:0 + (4  $\times$  C14:0) + C16:0]/( $\Sigma$  MUFA +  $\Sigma$  PUFA).

Spreadability index = (C16:0/C18:1).

Ni = pasture-fed cream; Ci = control Italian cream; Cb: control Belgian cream; Nb= Belgian farm cream.

approximately 75 % of the total fatty acids, consistent with common profiles in bovine milk fat (Lindmark Månsson, 2008). Short-chain saturated fatty acids, from C4:0 (butyric acid) to C8:0 (caprylic acid), as well as lauric acid (C12:0) and myristic acid (C14:0), were not statistically different between samples. The pasture-fed cream (Ni) had lower quantities of palmitic acid (C16:0) and stearic acid (C18:0), which led to a lower amount of saturated fatty acids (SFA). Furthermore, Ni exhibited higher amounts of monounsaturated FAs, such as oleic, myristoleic, and trans-11 vaccenic acid, as well as a higher amount of polyunsaturated fatty acids (Table 2). Similar trends in SFA, MUFA, and PUFA content were reported by Smet et al. (2010) in milk fat from cows fed extruded linseed, which also influenced ice cream characteristics. Pasture-based diets and high forage-to-concentrate ratios improve the fatty acid profile by increasing the quantity of unsaturated fatty acids and decreasing that of SFA in milk fat (O'Callaghan et al., 2017; O'Callaghan et al., 2016; Riuzzi et al., 2021).

Higher levels of rumenic acid, the main CLA isomer in ruminant fat, were observed in both Nb and Ni cream samples. In the Nb group, cows were fed a mix of fresh and silage forages with a forage-to-concentrate ratio of 80:20, compared to conventional TMR diets, which typically use a 60:40 ratio or lower. Rumen biohydrogenation of dietary unsaturated fatty acids, such as linoleic acid, leads to the formation of CLA and stearic acid. The latter can be desaturated back into CLA in the mammary gland via stearoyl-CoA desaturase, an enzymatic activity shown to be more pronounced in pasture-fed animals (Tudisco et al., 2019). VA and CLA isomers are linked to beneficial health effects in humans, including anti-inflammatory and immunomodulatory properties (Balivo et al., 2023).

The higher MUFA and PUFA content relative to SFA can influence milk fat properties due to their lower melting points. Couvreur et al. (2006) proposed the palmitic-to-oleic acid ratio (C16:0/C18:1) as a spreadability index for butterfat. In this study, Ni showed the lowest index (1.31), and Ci the highest (1.73) (Table 2). Ni also had significantly higher levels of branched-chain fatty acids (BCFAs), which increase with fresh pasture intake and are associated with lower melting points and softer fat (Vlaeminck et al., 2006). These spreadability index values fall within the range typically reported for milk fat (0.6–3) (Timlin et al., 2024), suggesting a slightly softer fat in Ni. However, this index should be interpreted as an indicator of relative differences in FA composition rather than an absolute measure of spreadability, since fat texture is also affected by crystallization kinetics, solid fat content profile and the size of milk fat globules (Danthine, 2012; Truong et al., 2016). Moreover, fatty acid composition was determined on the cream used as the ice cream ingredient and source of fat, but freezing and storing ice cream could potentially alter the fatty acid profile, and

further investigation evaluating these effects would be interesting.

Cooling profiles were characterised by two exothermic events (Fig. 1). The first crystallization peak, at about 11–12 °C, was the minor peak in terms of energy. The second peak, at approximately 14–16 °C, represented the major one in amount of energy. Melting profiles were characterized by three endothermic peaks: one with a maximum at about 2 °C, corresponding to the low melting fraction (LMF), another at 14–15 °C, the major melting peak in energy, corresponding to the medium melting fraction (MMF), and the final peak at 37–38 °C, corresponding to the high melting fraction (HMF) (Tomaszewska-Gras, 2013). Overall, Ni cream had a lower melting and crystallization enthalpy, and a lower final melting temperature (Table 3). Couvreur et al. (2006) reported that replacing maize silage with increasing proportions of fresh grass (0–100 %) led to a linear decrease in the final melting temperature, melting enthalpy, and solid fat content of milk fat. This effect is attributed to the enrichment of low-melting fatty acids, which reduce both the enthalpy and temperature of the final melting peak associated with high-melting triglycerides (Schäffer et al., 2001). Similarly, Bazmi and Relkin (2006) observed a lower final melting temperature and melting enthalpy for the bulk fat composed of a mixture (2:1) of anhydrous milk fat and LMF of the milk fat compared to the samples represented by anhydrous milk fat or anhydrous milk fat mixed (2:1) with the HMF.

Ci showed the highest  $T_{con}$  and crystallization enthalpy, followed by Nb and Cb, and then Ni, with the lower enthalpy mainly due to the lower energy of the second crystallization peak (Table 3), as observed by Schäffer et al. (2001) with the enrichment of milk fat with 25 % LMF. The lower crystallized fraction in Ni was also reflected in its SFC, where Ni generally showed a lower value at each temperature (Fig. 2). This aligns with previous studies showing that replacing conserved forages with fresh pasture reduces SFA and SFC in milk fat (Couvreur et al., 2006; Zhao et al., 2023). Murphy et al. (1995) found that diets increasing oleic acid content led to lower SFC at 5 and 10 °C. Reduced SFC, particularly in olein-rich fractions, has been associated with lower firmness at both room and refrigerated temperatures, affecting spreadability and functionality of milk fat (Queiros et al., 2016).

These findings highlight how pasture-based diets affect the fatty acid composition and physical properties of cream, offering insights into its functionality in products like ice cream.

### 3.2. Thermal behaviour and solidified fat content of ice cream mix

The aged Ni ice cream mix exhibited the lowest crystallization enthalpy, while Ci showed the highest, followed by Nb and Cb (Table 4), in agreement with the results observed in the corresponding cream fat extracts (Table 3). This trend is consistent with the higher content of SFAs in Ci > Cb > Nb > Ni, as SFAs crystallize at higher temperatures than unsaturated FAs. The results also indicate a shift towards lower crystallization temperature peaks for fat in emulsion within the ice cream mixes compared to the bulk fat phase. This supercooling effect was similar for Ni, Ci, Cb and Nb emulsions (Fig. 3). However, as expected for emulsions containing  $\approx$ 10 % milk fat, the crystallization and melting enthalpies were relatively small compared to the bulk fat.

Similar displacements of the main crystallization event in emulsified systems have been previously reported (Barford et al., 1991; Granger et al., 2005; Zhao et al., 2023). This delayed crystallization in emulsions is due to different nucleation mechanisms. In emulsified systems, homogeneous nucleation occurs within the numerous dispersed fat droplets, while in bulk fat, heterogeneous nucleation on impurities or surfaces leads to faster crystallization (Coupland, 2002; Mura & Ding, 2021).

On average, Ni had a lower final melting temperature (Table 4), likely due to its higher content of BCFAs as well as unsaturated FAs (Table 2), as also reflected in the thermal behaviour of the creams (Table 3). In 10 % milk fat ice cream model emulsions developed with anhydrous milk fat alone or blended (2:1) with high or low melting

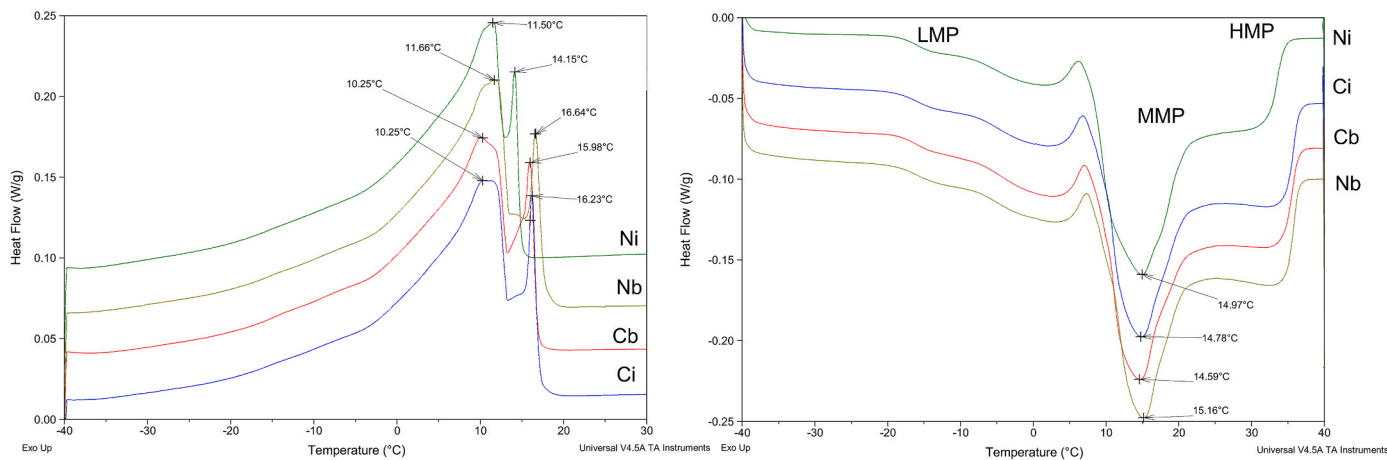


Fig. 1. Crystallization (left) and melting (right) profiles of the fat extracts of the creams used for the production of ice cream.

**Table 3**  
Thermal properties (2 °C min<sup>-1</sup>) of the milk fat from creams used for ice cream production.

Sample	Crystallization				Melting				
	T <sub>Ccon</sub> (°C)	T <sub>C1</sub> (°C)	T <sub>C2</sub> (°C)	ΔH <sub>C</sub> (J/g)	T <sub>M1</sub> (°C)	T <sub>Mon</sub> (°C)	T <sub>M2</sub> (°C)	T <sub>Mend</sub> (°C)	ΔH <sub>M</sub> (J/g)
Ni	14.86 ± 0.04 d	14.16 ± 0.04 d	11.60 ± 0.11 a	66.12 ± 0.39 c	1.78 ± 0.08 c	7.49 ± 0.08 b	14.90 ± 0.05 b	37.27 ± 0.36 b	84.37 ± 2.13 b
Ci	17.81 ± 0.16 a	16.67 ± 0.05 a	11.74 ± 0.08 a	70.17 ± 0.45 a	2.72 ± 0.07 a	8.76 ± 0.44 a	15.31 ± 0.13 a	38.00 ± 0.16 a	86.77 ± 0.62 ab
Nb	17.23 ± 0.24 b	16.21 ± 0.05 b	11.19 ± 0.63 a	68.38 ± 0.58 b	2.22 ± 0.13 b	8.57 ± 0.23 a	14.97 ± 0.11 b	38.29 ± 0.34 a	87.41 ± 1.30 a
Cb	16.81 ± 0.09 c	15.94 ± 0.05 c	11.76 ± 0.03 a	67.57 ± 0.69 b	2.66 ± 0.10 a	8.95 ± 0.03 a	14.63 ± 0.03 c	38.54 ± 0.04 a	89.04 ± 1.79 a

Values are reported as mean followed by standard deviation (n = 3). Different letters for the same column indicate statistically significant differences (p < 0.05). Ni = pasture-fed cream; Ci = control Italian cream; Cb= control Belgian cream; Nb= Belgian farm cream.

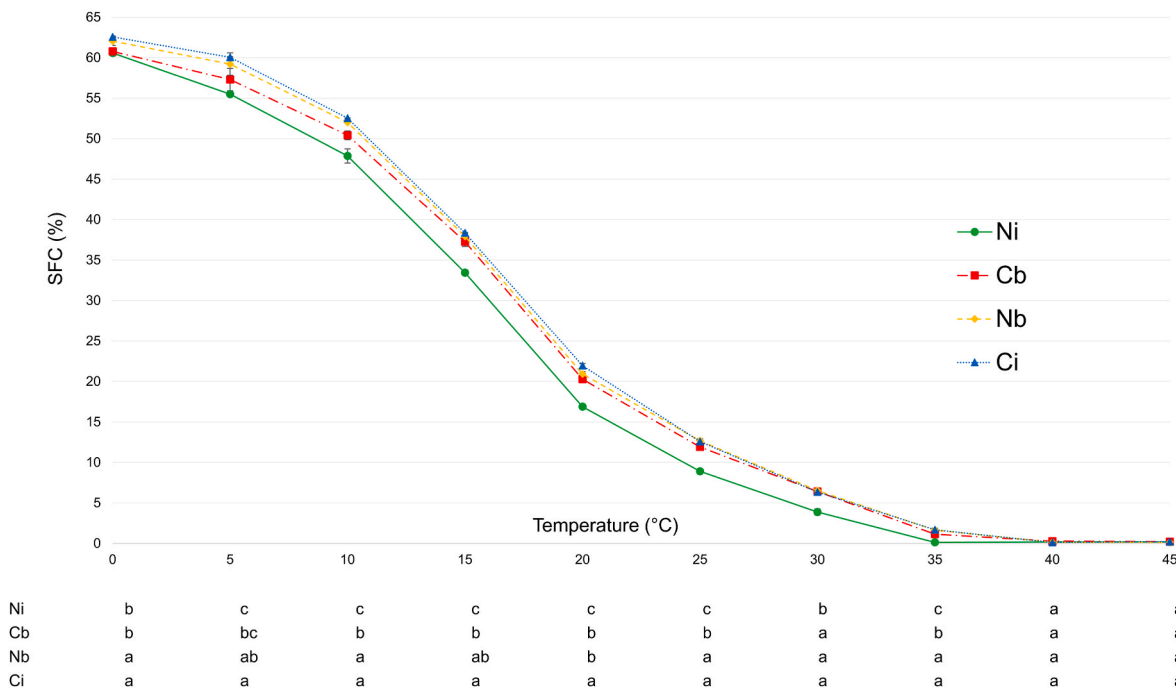


Fig. 2. Solid fat content (SFC) at different temperatures of fat extracts from creams used in the production of ice cream. Different letters of the same columns correspond to significant differences (p < 0.05).

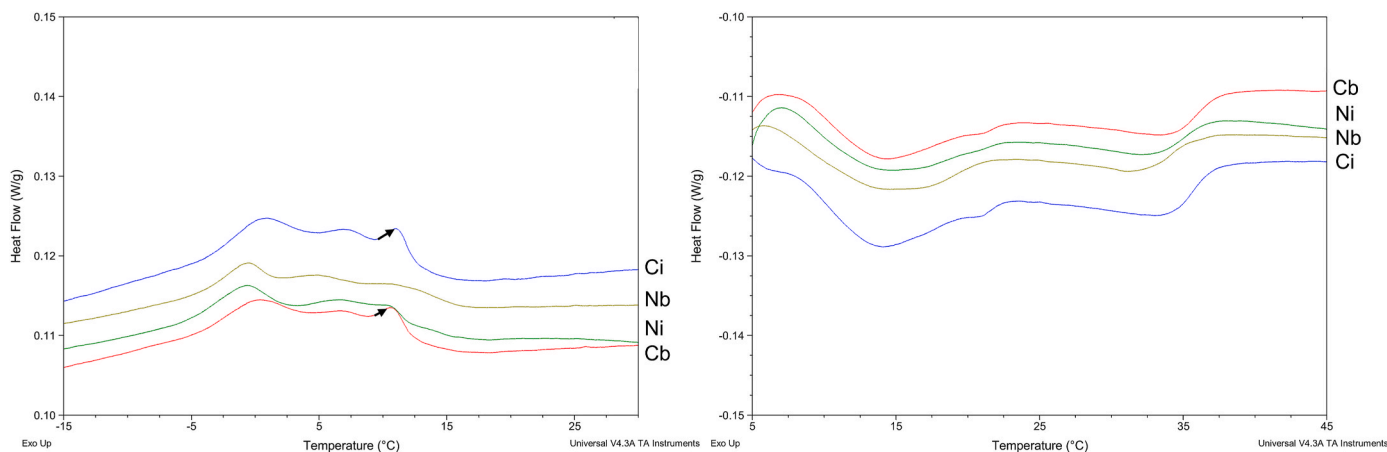
temperature milk fat fractions, it was reported a lower peak temperature of crystallization and a lower final melting temperature for the samples developed with the LMF, which they associated with a lower ratio of saturated to unsaturated FAs triglycerides in these samples (Bazmi et al., 2008; Bazmi & Relkin, 2006).

The physical state of fat plays a key role during mix aging and in the development of ice cream structure. As the mix cools to 4 °C after homogenization, fat droplets undergo partial crystallization, which is essential to enable partial coalescence during the subsequent dynamic freezing step, contributing to the fat network responsible for shape

**Table 4**  
Effect of cream type on the thermal properties ( $2\text{ }^{\circ}\text{C min}^{-1}$ ) of ice cream mix after aging.

Sample mix	Crystallization				Melting		
	T <sub>C</sub> on (°C)	T <sub>C</sub> max (°C)	ΔH <sub>C</sub> (J/g)	T <sub>M</sub> on (°C)	T <sub>M</sub> max (°C)	T <sub>M</sub> end (°C)	ΔH <sub>M</sub> (J/g)
Ni	13.29 ± 0.21 a	-0.59 ± 0.11b	3.24 ± 0.18 c	6.78 ± 0.30 a	14.38 ± 0.14 a	37.21 ± 0.68 c	3.48 ± 0.47 a
Ci	13.13 ± 0.21 a	0.67 ± 0.19 a	4.39 ± 0.12 a	7.74 ± 0.58 a	14.39 ± 0.33 a	39.42 ± 0.30 a	4.95 ± 1.13 a
Nb	13.71 ± 0.53 a	-0.27 ± 0.56ab	4.04 ± 0.18ab	7.91 ± 0.12 a	14.56 ± 0.20 a	37.83 ± 0.23 bc	4.40 ± 0.32 a
Cb	12.84 ± 0.21 a	0.59 ± 0.47 a	3.83 ± 0.10 b	7.37 ± 1.51 a	14.41 ± 0.06 a	39.16 ± 0.53 ab	4.50 ± 0.28 a

Values are reported as mean followed by standard deviation. Different letters for the same column indicate statistically significant differences ( $p < 0.05$ ).

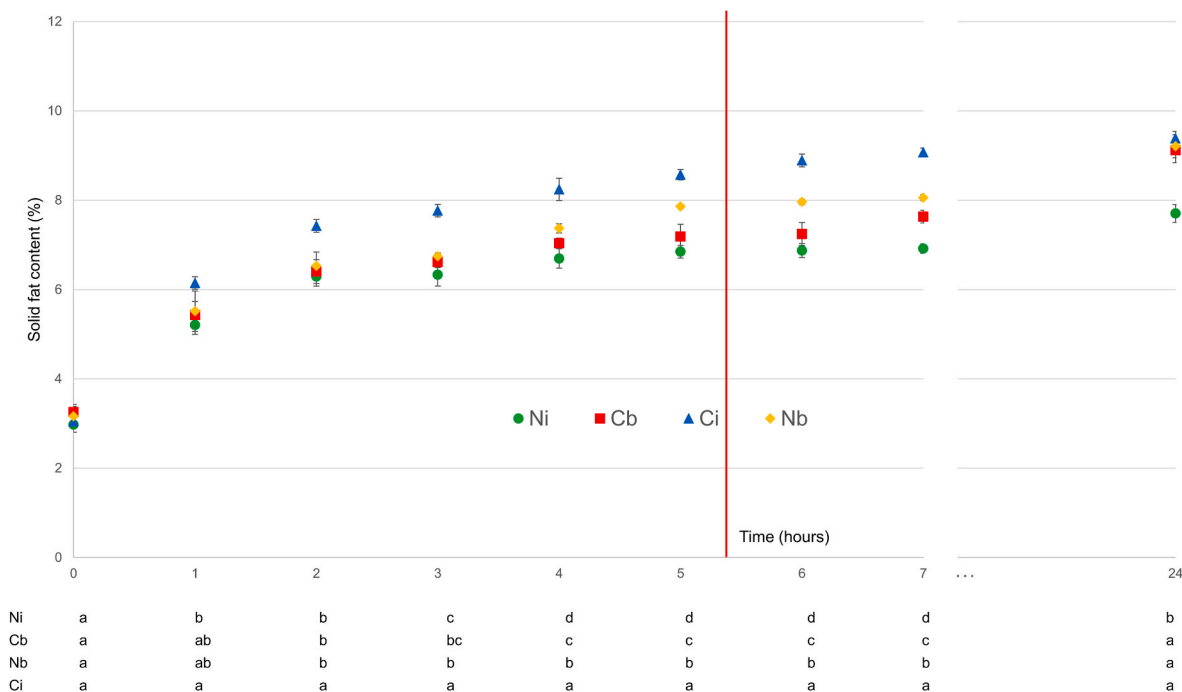


**Fig. 3.** Crystallization (left) and melting (right) profiles of ice cream mix emulsions after aging as affected by the cream type.

retention and melting resistance (D. H. Goff & R. W. Hartel, 2013). Fig. 4 shows the effect of cream type on solid fat content in the mixes over 24 h at 4 °C. Most crystallization occurs within the first 4–5 h, followed by a slower increase over time (Barford et al., 1991; D. H. Goff & R. W. Hartel, 2013). On average, the Ni mix samples exhibited a lower SFC during the ageing phase and at the end of ageing (after 24 h at 4 °C), prior to ice cream manufacturing. This can be attributed to their higher

content of MUFA and PUFA, as well as branched-chain FAs.

These branched-chain FAs have lower melting points, leading to a higher proportion of triacylglycerols remaining in liquid form (D. H. Goff & R. W. Hartel, 2013). This aligns with the lower crystallization enthalpy observed in mixes made with pasture-fed cream (Table 4), suggesting reduced triglyceride crystallization compared to control samples. Sung and Goff (2010) found that mixes formulated with fats



**Fig. 4.** The effect of cream type on the crystallization kinetics of the milk fat phase in ice cream mix emulsions during aging at 4 °C for 24 h. The vertical line indicates the “first phase” (until 4–5h), where most of the crystallization occurs.

containing a higher proportion of SFAs (hard fraction of palm kernel oil) compared to unsaturated ones (provided by high-oleic sunflower oil) exhibited a higher solid fat content. Abd El-Rahman et al. (1997) reported that the ice cream mix made with the addition of the low melting point milk fat fraction had lower SFC values at 5 °C both initially and after 24 h, with average SFC values at the end of the ageing period of 5.4 % compared to 9.4 % for the control cream mix. In contrast, mixes developed with the high melting point milk fat fraction showed an SFC value of 11.9 %. Similarly, Smet et al. (2010) found that milk fat enriched in unsaturated fatty acids, via dietary oilseeds or low-melting fractions, had reduced SFC under simulated processing conditions, supporting our results.

### 3.3. Rheological properties of ice cream mix

All samples exhibited non-Newtonian shear-thinning behaviour, consistent with previous studies (Goff et al., 1995; Marín-Suárez et al., 2016; Mortazavian et al., 2020; Trivana et al., 2023). Viscosity decreased more rapidly for the Ni mix in the initial shear rate ramp, stabilising at values similar to those of the Nb mix for shear rates above 10 s<sup>-1</sup>. Ni had a lower consistency index (K) and higher flow behavior index (n), and, along with Nb, showed lower apparent viscosity at 30 and 50 s<sup>-1</sup> (Table 5). In contrast, Ci exhibited the highest viscosity, with values of 0.97 Pa s at 30 s<sup>-1</sup> and 0.76 Pa s at 50 s<sup>-1</sup>. These shear rates were chosen because they are considered to simulate shear rates experienced in the mouth during food consumption (Chen, 2009; Mortazavian et al., 2020). An increased proportion of sunflower oil relative to coconut oil in the mixes led to a reduction in viscosity, with differences among samples diminishing at higher shear rates (Trivana et al., 2023). Gonzalez et al. (2003) found that the apparent viscosity of the ice cream mixes developed using butter enriched with unsaturated fatty acids (oleic and linoleic FAs), thanks to the integration of oilseeds into the cow's diet, was approximately 40 % lower compared to the control mix. Similarly, Nadeem, Abdullah, et al. (2015) observed lower viscosity after 24 h of aging at 4 °C in mixes using milk fat enriched in oleic acid through supplementation of calcium salts of fatty acids in the cow's diet. In line with Adapa et al. (2000), we observed a slight increase in the storage modulus (G') and loss modulus (G'') with increasing frequency (Table 5). At 0.5 Hz, the Ci sample showed the highest G' (35.40 Pa), while Ni had the lowest (21.00 Pa); a similar trend was observed for G''.

**Table 5**

The effect of cream type on the rheological properties of ice cream mix at 4 °C after ageing.

Mix	Viscous properties					
	Yield stress $\tau_0$ (Pa)	consistency index K (Pa·s <sup>n</sup> )	flow behavior index n	Apparent viscosity at 30 s <sup>-1</sup>	Apparent viscosity at 50 s <sup>-1</sup>	R <sup>2</sup> <sup>a</sup>
Ni	0.46 ± 0.07 a	1.91 ± 0.45 b	0.68 ± 0.06 a	0.64 ± 0.04 bc	0.54 ± 0.04 bc	0.996 ± 0.002
Ci	0.49 ± 0.08 a	4.67 ± 0.70 a	0.53 ± 0.02 b	0.97 ± 0.09 a	0.76 ± 0.06 a	0.998 ± 0.001
Nb	0.49 ± 0.00 a	3.56 ± 0.33 a	0.46 ± 0.02 b	0.58 ± 0.11 c	0.43 ± 0.08 c	0.997 ± 0.004
Cb	0.48 ± 0.12 a	4.32 ± 0.44 a	0.46 ± 0.04 b	0.75 ± 0.05 b	0.56 ± 0.04 b	0.998 ± 0.002
Mix	Viscoelastic properties					
	Frequency (Hz)	Storage modulus G' (Pa)	Loss modulus G'' (Pa)	Damping factor tan $\delta$	Complex viscosity, $\eta^*$ (Pa·s)	
Ni	0.5	21.00 ± 2.23 c	9.48 ± 0.89 c	0.45 ± 0.02 a	7.33 ± 0.75 c	
Ci	0.5	35.40 ± 1.41 a	14.10 ± 0.42 a	0.41 ± 0.00 a	12.15 ± 0.49 a	
Nb	0.5	24.37 ± 1.08 c	10.53 ± 0.06 c	0.43 ± 0.02 a	8.43 ± 0.30 c	
Cb	0.5	30.20 ± 0.95 b	12.77 ± 0.46 b	0.42 ± 0.00 a	10.40 ± 0.35 b	
Ni	1	25.10 ± 3.16 c	9.31 ± 0.89 c	0.37 ± 0.01 a	4.27 ± 0.52 c	
Ci	1	41.80 ± 0.71 a	14.10 ± 0.42 a	0.34 ± 0.00 b	7.02 ± 0.13 a	
Nb	1	29.03 ± 1.89 c	10.14 ± 0.19 c	0.35 ± 0.02 ab	4.90 ± 0.29 c	
Cb	1	35.00 ± 1.14 b	11.80 ± 0.44 b	0.34 ± 0.00 b	5.88 ± 0.19 b	
Ni	2	24.40 ± 1.50 c	8.93 ± 0.68 c	0.33 ± 0.06 a	2.36 ± 0.52 b	
Ci	2	44.80 ± 1.70 a	13.40 ± 0.42 a	0.30 ± 0.02 a	3.73 ± 0.13 a	
Nb	2	27.70 ± 0.30 c	9.32 ± 0.45 c	0.30 ± 0.04 a	2.62 ± 0.51 ab	
Cb	2	34.50 ± 1.20 b	10.70 ± 0.26 b	0.31 ± 0.00 a	2.88 ± 0.10 ab	

Values are reported as mean followed by standard deviation. Different letters for the same column indicate statistically significant differences (p < 0.05).

<sup>a</sup> R<sup>2</sup> values are the coefficients of determination for the Herschel-Bulkley model used to fit the experimental data.

Overall, the damping factor (tan  $\delta$ ), indicative of viscoelastic behaviour, did not differ significantly among samples.

### 3.4. Fat globule distribution in mix and ice cream

The fat particle size in the mixes was measured after homogenization to determine the initial particle size distribution before freezing and to explore fat destabilization in ice cream. The mean size volume diameter of the fat particles ranged from 1.48 to 1.69  $\mu$ m across the studied emulsion mixes (Table 6). In ice cream samples, a bimodal distribution was observed: one peak appeared at approximately 1.5  $\mu$ m, corresponding to individual fat droplets, and an additional peak emerged in the range of 5–10  $\mu$ m, indicating aggregated fat clusters due to particle coalescence (Fig. 5). The mean particle diameter values for ice cream after production ranged from 4.09 to 5.07  $\mu$ m and slightly increased during storage (Table 6).

The Ci ice cream exhibited a greater increase in particle diameter due to more pronounced fat agglomeration, while the Ni and Nb ice creams showed a smaller increase. Bazmi et al. (2008) previously reported slightly smaller particles in ice cream mix emulsions (10 % fat) prepared with milk fat enriched in the olein fraction, compared to those made with control milk fat or milk fat enriched in the stearin fraction. Sung and Goff (2010) reported that the higher SFC of the mix, as observed in Ci (Fig. 4), resulted in a higher rate of fat destabilization. Similarly, Smet et al. (2010) found that the milk fat with a higher SFC resulted in large particles and a high content of aggregates in ice cream. This was due to the agglomeration of partially crystallized fat droplets, which were held together by fat crystals and liquid fat as long as the crystalline fat had not melted (Bazmi et al., 2008).

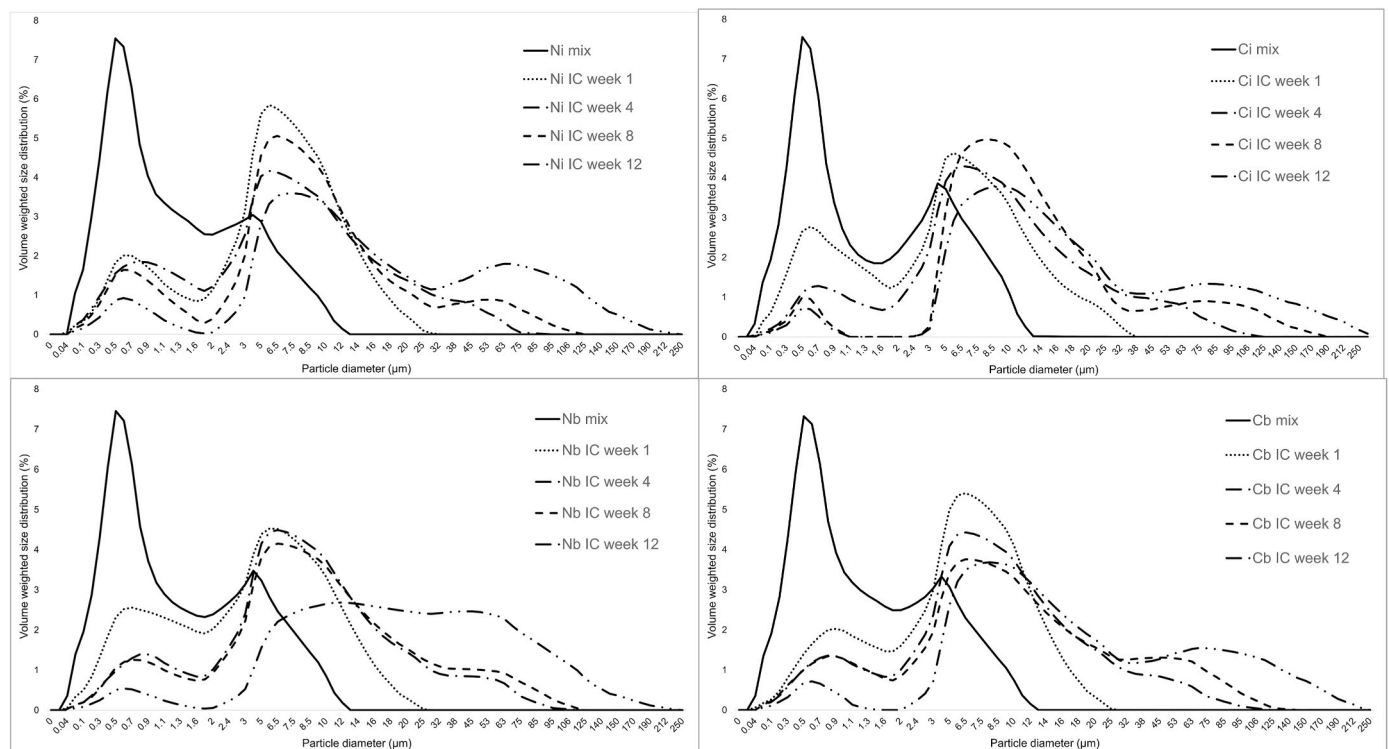
### 3.5. Overrun and melting properties of ice cream

The overrun values, indicating the amount of air incorporated during ice cream production, did not differ significantly among samples, ranging from 30.17 ± 1.68 % to 32.19 ± 4.29 %. Similarly, no significant differences in overrun values of ice cream were observed when using cream, anhydrous milk fat, and milk fat enriched with fractions with different melting points (Smet et al., 2010). Vargas-Bello-Pérez et al. (2019) found a lower overrun in ice creams made with cream from cows supplemented with fish oil, but not with soybean oil, despite both

**Table 6**  
Effect of cream type on fat particle size parameters in mixes and ice creams, and on the melting rate of ice creams.

Mix	Storage week	Mean size volume D <sub>[4,3]</sub> (µm)	Mean size surface D <sub>[3,2]</sub> (µm)	D90 vol (µm)	Fat destabilization % *	Melting rate (g/min)	First dripping time (min)	Complete melting time (min)
Ni		1.48 ± 0.01 c				0.40 ± 0.01 a		4.06 ± 0.03 d
Ci		1.69 ± 0.03 a				0.32 ± 0.01 b		4.82 ± 0.07 a
Nb		1.53 ± 0.02 b				0.31 ± 0.01 c		4.34 ± 0.02 b
Cb		1.49 ± 0.02 c				0.31 ± 0.01 c		4.17 ± 0.01 c
Ice cream	Storage week	Mean size volume D <sub>[4,3]</sub> (µm)	Mean size surface D <sub>[3,2]</sub> (µm)	D90 vol (µm)	Fat destabilization % *	Melting rate (g/min)	First dripping time (min)	Complete melting time (min)
Ni	1	5.07 ± 0.05 a	0.93 ± 0.05 a	10.47 ± 0.10 a	157 ± 0.93 a	0.60 ± 0.02 a	5.09 ± 0.10 a	60.6 ± 0.44 c
Ci	1	4.63 ± 0.02 b	0.72 ± 0.02 b	10.71 ± 0.11 a	122 ± 4.58 b	0.52 ± 0.01 b	6.80 ± 0.09 a	63.6 ± 0.43 ab
Nb	1	4.09 ± 0.15 c	0.72 ± 0.03 b	9.47 ± 0.20 b	118 ± 4.59 b	0.53 ± 0.02 b	6.22 ± 0.61 a	62.4 ± 0.12 b
Cb	1	5.02 ± 0.27 a	0.98 ± 0.08 a	10.37 ± 0.31 a	148 ± 7.60 a	0.52 ± 0.01 b	6.28 ± 1.14 a	64.9 ± 1.07 a
Ni	4	7.65 ± 0.74 b	1.01 ± 0.09 c	17.51 ± 1.87 b		0.58 ± 0.01 a	6.76 ± 0.30 a	62.3 ± 0.32 ab
Ci	4	10.68 ± 0.59 a	1.23 ± 0.03 b	24.85 ± 1.84 a		0.53 ± 0.01 b	5.25 ± 0.31 b	61.4 ± 0.37 b
Nb	4	9.62 ± 0.52 a	1.45 ± 0.02 a	21.01 ± 1.49 ab		0.53 ± 0.02 b	6.38 ± 0.63 ab	62.4 ± 0.53 ab
Cb	4	9.66 ± 1.21 a	1.38 ± 0.13 ab	21.44 ± 3.32 ab		0.51 ± 0.03 b	6.28 ± 0.75 ab	62.8 ± 0.69 a
Ni	8	9.96 ± 0.51 d	1.23 ± 0.05 b	20.46 ± 1.79 d		0.61 ± 0.01 a	5.04 ± 0.17 a	60.3 ± 0.25 c
Ci	8	16.83 ± 0.32 a	1.86 ± 0.05 a	40.58 ± 0.25 a		0.53 ± 0.01 b	6.53 ± 0.26 a	62.1 ± 0.16 b
Nb	8	11.42 ± 0.25 c	1.31 ± 0.16 b	27.81 ± 0.86 c		0.53 ± 0.02 b	5.45 ± 1.15 a	63.8 ± 0.36 a
Cb	8	13.09 ± 0.38 b	1.38 ± 0.15 b	36.30 ± 0.50 b		0.53 ± 0.01 b	6.52 ± 1.03 a	63.3 ± 0.40 a
Ni	12	24.41 ± 1.46 b	1.82 ± 0.12 b	72.36 ± 2.99 b		0.56 ± 0.01 a	9.32 ± 0.48 a	63.4 ± 0.60 b
Ci	12	33.63 ± 0.46 a	2.60 ± 0.07 a	96.57 ± 1.58 a		0.51 ± 0.01 b	11.02 ± 0.85 a	65.5 ± 0.68 a
Nb	12	24.49 ± 0.44 b	2.45 ± 0.32 a	59.23 ± 1.84 c		0.53 ± 0.00 b	10.61 ± 0.77 a	65.1 ± 0.16 a
Cb	12	26.40 ± 0.74 b	2.26 ± 0.06 ab	77.54 ± 2.45 b		0.54 ± 0.00 b	10.25 ± 0.90 a	64.4 ± 0.43 ab

Values are reported as mean followed by standard deviation. Different letters for the same column and storage time indicate statistically significant differences ( $p < 0.05$ ). The melting rate (g/min) was determined as the slope of the linear regression line. \*Percentage of particles with mean diameters greater than the cumulative volume distribution at the 90th percentile of the mix.



**Fig. 5.** The effect of cream type on the mean values of fat particle size distribution in mix and ice cream samples during storage.

diets providing similar fatty acid profiles of milk fat with higher unsaturated fatty acids compared to the control. However, the fish oil supplementation led to a higher amount of C20:3n-3 and C20:3n-6 fatty acids, which might have caused lower efficiency in retaining air in the matrix. In contrast, [Corradini et al. \(2014\)](#) did not obtain differences in overrun values in the case of milk fat obtained from animals

supplemented with different vegetable oils. Generally, overrun differences tend to be more evident when ice cream formulations contain very high levels of unsaturated fatty acids, as observed by [Marín-Suárez et al. \(2016\)](#).

After production, Ni ice creams exhibited the highest melting rate (0.60 g/min). On average, the melting rate tended to decrease during

storage, particularly after 3 months (Table 6). Overall, differences in the time to first drip were not significant, although Ni ice creams melted completely in a shorter time. Ice creams made with milk fat having a higher SFC and a higher melting point due to a greater amount of SFAs exhibited a greater resistance to melting (Abd El-Rahman et al., 1997; Smet et al., 2010). Soybean and fish oil supplementation in the cow's diet increased the unsaturated and reduced the saturated FA content of milk fat, resulting in higher ice cream melting rates of 0.87 and 0.94 g/min, respectively, compared to 0.62 g/min in the control (Vargas-Bello-Pérez et al., 2019). Similarly, Ni had a higher amount of unsaturated and branched-chain FAs (Table 2), which lower the melting temperature of milk fat. Furthermore, melting properties are also correlated with fat particle destabilization and aggregation during storage. As previously reported, fat agglomeration rate was generally lower for Ni ice creams during storage (Table 6). The higher SFC of the mix led to a higher rate of fat destabilization and agglomeration, resulting in a slower melting rate of the ice cream (Sung & Goff, 2010). Méndez-Velasco and Goff (2011) and Muse and Hartel (2004) also found that greater fat agglomeration in ice creams was associated with slower melting rates. The agglomeration of fat can form a network that surrounds the air cells, thereby increasing resistance to meltdown.

### 3.6. Texture and colour of ice cream

After production, no significant differences were observed among the samples ( $p > 0.05$ ), despite a trend towards lower hardness for Ni (Table 7). This aligns with Gonzalez et al. (2003), who reported similar results in ice creams made with milk fat from cows supplemented with high oleic and high linoleic safflower oil. An increase in hardness was observed for all samples, particularly after 2 and 3 months of storage. Additionally, significant differences in hardness were found, with Ni and Nb showing lower values compared to Cb and Ci.

The increase in hardness was also correlated with greater fat destabilization during storage, where destabilized fat forms a network around air cells in the ice cream, thereby increasing hardness (Muse & Hartel, 2004). In this study, Ni and Nb showed the lowest levels of fat agglomeration and destabilization over time (Table 6), which likely contributed to their lower hardness values. This relationship

underscores the influence of fat structure and stability on the texture of ice cream during its storage. Although no sensory analysis was conducted in this study, previous research has shown that instrumental differences in hardness do not always correspond to perceptible sensory differences in texture (Vargas-Bello-Pérez et al., 2019). Moreover, the higher SFC, in addition to resulting in a higher rate of fat destabilization and agglomeration, was associated with slower melting rates (Muse & Hartel, 2004).

Ni exhibited lower lightness ( $L^*$ ) and higher yellowness ( $b^*$ ) than the control samples (Ci and Cb), as also reflected by its lower whiteness index (WI) (Table 7). The Nb sample showed the highest  $b^*$  among all samples analysed, likely due to the cows' diet, which consisted of 80 % forage, including maize silage and fresh forage. These feeds, in contrast to hay and the concentrate portion of the diet, are known to contain higher levels of carotenoids, which may contribute to a more yellow colour in the final product. These differences were evident in both the mixes and the ice creams analysed over the storage weeks. In particular, Ni consistently showed lower  $L^*$  and higher  $b^*$  values, suggesting that pasture-fed cream may contribute to a yellower and less bright tone. Gonzalez et al. (2003) observed more pronounced colour differences after 3 and 5 months of storage, particularly noting higher yellowness in butter and ice cream from cows fed with maize silage compared to those supplemented with safflower oil.

## 4. Conclusions

This study demonstrated that pasture-fed cream, with higher levels of unsaturated and branched-chain fatty acids, influences the physical properties of ice cream by reducing solid fat content, melting temperature, and crystallization enthalpy. The resulting ice cream mixes showed lower apparent viscosity and reduced viscoelastic moduli ( $G'$  and  $G''$ ).

The melting rate tended to decrease during ice cream storage, while hardness increased, particularly after 3 months. This was associated with greater fat destabilization, leading to the formation of a fat network around air cells in the ice cream. However, given the limited literature on fat coalescence during storage, microscopic confirmation would be necessary to verify whether the observed increase truly reflects partial fat coalescence.

**Table 7**  
Effect of cream type on ice cream hardness and colour during storage.

Sample	Storage week	$L^*$	$a^*$	$b^*$	WI	
<b>Mix</b>						
Ni		92.4 ± 0.02 ab	-1.54 ± 0.03 b	12.6 ± 0.03 b	85.2 ± 0.04 b	
Ci		92.2 ± 0.02 b	-1.15 ± 0.06 a	11.0 ± 0.15 c	86.5 ± 0.12 a	
Nb		90.7 ± 0.05 c	-1.14 ± 0.03 a	15.2 ± 0.11 a	82.2 ± 0.12 c	
Cb		92.8 ± 0.06 a	-1.41 ± 0.04 b	10.4 ± 0.18 c	87.13 ± 0.21 a	
<b>Ice cream</b>						
	Storage week	$L^*$	$a^*$	$b^*$	WI	Hardness (N)
Ni	1	92.6 ± 0.14 a	-1.50 ± 0.07 b	12.2 ± 0.13 b	85.7 ± 0.13 b	22.8 ± 3.55 a
Ci	1	92.8 ± 0.02 a	-2.02 ± 0.05 c	10.2 ± 0.21 c	87.4 ± 0.28 a	29.6 ± 2.58 a
Nb	1	91.4 ± 0.05 b	-1.09 ± 0.07 a	14.8 ± 0.05 a	82.8 ± 0.02 c	23.5 ± 3.72 a
Cb	1	92.9 ± 0.11 a	-1.73 ± 0.13 bc	10.9 ± 0.22 c	86.9 ± 0.29 a	27.7 ± 3.61 a
Ni	4	92.5 ± 0.16 b	-1.09 ± 0.05 b	11.5 ± 0.10 b	86.2 ± 0.16 c	22.1 ± 3.30 b
Ci	4	92.8 ± 0.06 a	-2.07 ± 0.05 d	10.0 ± 0.14 d	87.5 ± 0.07 a	30.1 ± 1.78 a
Nb	4	90.0 ± 0.01 c	-1.17 ± 0.01 a	14.7 ± 0.02 a	82.3 ± 0.01 d	24.0 ± 3.22 b
Cb	4	92.8 ± 0.07 a	-1.62 ± 0.03 c	10.9 ± 0.08 c	86.8 ± 0.09 b	28.7 ± 1.60 a
Ni	8	91.4 ± 0.02 b	-1.39 ± 0.02 a	12.2 ± 0.02 b	85.0 ± 0.03 b	30.3 ± 3.31 c
Ci	8	91.5 ± 0.02 b	-2.14 ± 0.02 b	10.8 ± 0.03 c	86.0 ± 0.02 a	41.2 ± 2.46 a
Nb	8	90.4 ± 0.01 c	-1.38 ± 0.03 a	14.3 ± 0.02 a	82.7 ± 0.01 c	35.7 ± 1.03 b
Cb	8	91.6 ± 0.08 a	-1.39 ± 0.16 a	10.9 ± 0.20 c	86.2 ± 0.26 a	41.9 ± 2.90 a
Ni	12	91.4 ± 0.02 c	-1.02 ± 0.05 a	11.6 ± 0.19 b	85.5 ± 0.15 c	35.6 ± 2.63 a
Ci	12	92.2 ± 0.03 a	-1.45 ± 0.03 c	9.5 ± 0.08 d	87.6 ± 0.07 a	49.5 ± 4.90 b
Nb	12	90.4 ± 0.01 d	-1.36 ± 0.02 b	14.1 ± 0.02 a	82.9 ± 0.03 d	38.7 ± 3.42 a
Cb	12	92.1 ± 0.03 b	-1.37 ± 0.03 b	10.9 ± 0.05 c	86.5 ± 0.04 b	49.2 ± 3.91 ab

Values are reported as mean followed by standard deviation. Different letters for the same column indicate statistically significant differences ( $p < 0.05$ ). WI: whiteness index; N: Newtons.

Further research could expand on these findings by investigating consumer sensory responses to ice creams made with pasture-fed cream, also in formulations incorporating additional flavouring agents (e.g., vanilla, cocoa).

### CRedit authorship contribution statement

**Andrea Balivo:** Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Alessandro Genovese:** Writing – review & editing, Supervision, Conceptualization. **Giorgia Purcaro:** Writing – review & editing, Resources. **Sabine Danthine:** Writing – review & editing, Supervision, Resources, Methodology, Conceptualization.

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### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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### Data availability

Data will be made available on request.

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