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UNIVERSIDAD AUTÓNOMA METROPOLITANA
UNIDAD IZTAPALAPA

ESTUDIO DE PROPIEDADES FISICOQUÍMICAS, TÉRMICAS Y SENSORIALES EN HELADOS REDUCIDOS EN GRASA Y AZÚCAR USANDO INULINA DE ACHICORIA Y FRUCTANOS DE AGAVE COMO REMPLAZO

Doctorado en Biotecnología

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Ciudad de México, 2019

México D.F. a 19 de Julio del 2019

El jurado designado por la

División de Ciencias Biológicas y de la Salud de la Unidad Iztapalapa aprobó la tesis

ESTUDIO DE PROPIEDADES FISICOQUÍMICAS, TÉRMICAS Y SENSORIALES EN HELADOS REDUCIDOS EN GRASA Y AZÚCAR USANDO INULINA DE ACHICORIA Y FRUCTANOS DE AGAVE COMO REMPLAZO

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El Doctorado en Biotecnología de la Universidad Autónoma Metropolitana está incluido en el Programa Nacional de Posgrados de Calidad (PNPC) del Consejo Nacional de Ciencia y Tecnología (CONACyT), con la referencia número 001466.

La primera parte de esta tesis se llevó a cabo en Laboratorio de Alimentos en el Tecnológico de Estudios Superiores de Ecatepec conjuntamente con la Unidad de Investigación Multidisciplinaria de la FES Cuautitlán, UNAM, Estado de México. Posteriormente la parte sensorial fue desarrollada en el Laboratorio de Análisis Sensorial y de Estudios con consumidor del Departamento en Biotecnología de la Universidad Autónoma Metropolitana de Iztapalapa conjuntamente con el Laboratorio de Evaluación Sensorial del Departamento de Alimentos y Biotecnología en la Facultad de Química, UNAM, Ciudad de México.

Esta tesis fue financiada con la beca número 246848 otorgada por el programa de becas a posgrado del CONACyT.

Agradecimientos

Quisiera agradecer a la Universidad Autónoma Metropolitana de Iztapalapa por la gran oportunidad de realizar mis estudios doctorales en su prestigiada institución ¡Un gran sueño para mí!. Especialmente al Dr. Hector por permitirme ser parte de su equipo de trabajo, por incluirme en sus actividades, por compartir su valiosa amistad y su tiempo, pero sobre todo por la confianza y el apoyo que me brindó… ¡Mil gracias Doc!

Por el apoyo y cariño de mis compañeros del laboratorio de sensorial y enología: Ara, Angélica, Pilar, Alfredo, Adriana, Mariel, Luis, Fernanda, Miguel, Dany, el Dr. Ramón y la Dra. Frida y por todas las personas bellas que conocí durante este periodo.

Gracias al Tecnológico de Estudios Superiores de Ecatepec por todo el apoyo desde mi licenciatura, especialmente al Dr. Alfonso Totosaus por guiar la primera parte del proyecto y del cual se obtuvo la primera publicación que tiene por título “Effect of inulin on melting and textural properties of low-fat and sugar-reduced ice cream: optimization via a response surface methodology”.

A la Unidad Multidisciplinaria de Investigación de la FES Cuautitlán de la UNAM, especialmente al Dr. Arjona por asesorarme en la parte de calorimetría y de la cual se obtuvo la segunda publicación que tiene por título “The influence of agave fructans on thermal properties in low-fat and low-fat and sugar ice cream”.

A la Dra. Patricia Severiano por sus revisiones y correcciones de los dos últimos artículos, por todo el apoyo durante el doctorado, y por su hermosa y valiosa amistad que tenemos desde hace unos años. A Mariana y a todos los jueces que brindaron los resultados para poder escribir la cuarta publicación que tiene por título “Temporal Dominance of Sensations (TDS) and Temporal check all that Apply (T-CATA) for the dynamic characterizing of different ice cream formulations”.

A la Dra. Paula Varela y la Dra. Susana Fiszman por sus revisiones y por abrirme las puertas e inquietarme a buscar más.

Gracias a mis sinodales la Dra. Angélica Román, la Dra. Gabriela Rodríguez y la Dra. Carmen Hernández por revisar mi tesis y hacer sus valiosos comentarios.

A mis padres Beatriz y Celso que siempre han sido una parte fundamental en mi formación, por su ejemplo, su apoyo y su amor... A mis hermanos Adan y Janet por siempre apoyarme y darme por mi lado, por siempre estar cuando los necesito… Gracias

A los güeritos y a Lupita por todo su apoyo durante este año, por todo su amor y paciencia hacia mi familia.

A Hugo por acompañarme en esta aventura, por su compromiso, paciencia, comprensión y tiempo, pero sobre todo por el amor que me ha demostrado. Especialmente quiero agradecer a mi hijo Gael por cambiar mi vida y demostrarme lo maravilloso que es ser madre. Gracias por darme fuerzas para seguir adelante y motivarme cada día…

Resumen

La tesis a lo largo de cuatro capítulos plantea el efecto que causa modificar la formulación de los helados, especialmente cuando compuestos tan importantes como son grasa y azúcar son fundamentales para mantener una microestructura deseada que se ve reflejada en una buena calidad.

En el primer capítulo se analizó el efecto que provoca el uso la inulina de achicoria sobre las propiedades de derretimiento y de textura cuando la grasa butírica y el azúcar son reducidos en el helado, usando como metodología una optimización de superficie de respuesta. Posteriormente el segundo capítulo da a conocer los resultados del uso de fructanos de agave sobre las propiedades térmicas en dos tipos de formulaciones: helados reducidos en grasa y helados reducidos en grasa y azúcar. Estas mismas formulaciones fueron usadas para realizar un análisis sensorial y de textura, resultados que fueron utilizados para el desarrollo del tercer capítulo, en el cual se analizó una triple correlación entre las propiedades sensoriales, térmicas y de textura, cuando grasa y azúcar fueron sustituidos por fructanos de agave. Finalmente, el cuarto capítulo muestra los resultados de la caracterización dinámica de diferentes marcas comerciales de helado, así como nuestras formulaciones reducidas, usando fructanos de agave.

El propósito general de la tesis tiene como objetivo servir de base para la re-formulación de helados y dar a conocer a los productores nuevas alternativas de compuestos que ayudan a mejorar las propiedades de los mismos. Así como implementar nuevas opciones para los consumidores de este producto.

Summary

Throughout four chapters of the thesis, we evaluated form different perspectives, the effect of reducing fat and/or sugar on the formulation of the ice creams, especially because these compounds are fundamental to maintain a desirable microstructure, which is reflected in a good quality of ice cream.

The first article analyzed the effect that provoke the use of chicory inulin about melting and texture properties when the butyric fat and sugar were reduce in ice cream, using an optimization via response surface methodology. Later, the second paper revels the results about the use of agave fructans about thermal properties in two group of formulations: low fat and low fat and sugar ice cream. Those same formulations were used to do a sensory and texture analysis, results that were used to develop the third publication, which studied the triple correlation among sensory, thermal and textural properties, when fat and sugar were replace for agave fructans. Finally, the fourth paper showed the results of the dynamic characterization of different brands of ice cream, as well as our reduced formulations, using agave fructans.

The general purpose of the thesis is to serve as a basis for the re-formulation of ice cream and to inform the producers of new alternatives of compounds that help improve their properties. As well as implement new options for consumers of this product.

Publicaciones

Pintor A, Escalona H. and Totosaus A. (2017). Effect of inulin on melting and textural properties of low-fat and sugar-reduced ice cream: optimization via a response surface methodology. *International Food Research Journal* **24** 1728-1734.

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Pintor A, Arjona L, Severiano P, Totosaus A, Fiszman S and Escalona H (2019). Agave fructans as fat and sugar replacer in ice cream: sensory, thermal and textural properties. (Food Hydrocolloids).

Pintor A, Luque M, Varela Paula, Escalona H. and Severiano-Pérez P. (2019). Characterization by dynamics methods of comercial and low fat and sugar ice cream using agave fructans as replacer (Dairy Food).

Publicación complementaria

Fragoso M, Pérez-Chabela M, Hernández A, Escalona H, **Pintor A**. and Totosaus A. (2016). Sensory, melting and textural properties of fat-reduced ice cream inoculated with thermotolerant lactic acid bacteria. *Carpathian Journal of Food Science and Technology* **8** 11-21.

Participación en congresos

(**2016**) Congreso internacional, IFT, presentación de poster “Fat and sugar reduction in ice cream employing inulin as fat and sugar replacer”. Chicago Illinois, USA.

(**2017**) Congreso internacional, Pangborn, presentación de poster “Relationship between sensory and thermal properties on low fat and low sugar and fat ice creams using agave fructans as replacer”. Providence RI, USA.

(**2018**) Congreso internacional, Eurosense, presentación de poster “Correlation between sensory, thermal and textural properties on low fat and low fat and sugar ice creams using agave fructans as replacer”. Verona, Italia.

Índice

1. Introducción y Justificación

1.1 Introducción

Actualmente la industria de los alimentos se ha centrado en producir productos más sanos o con carga calórica reducida, debido a que los consumidores se inclinan cada vez más por el consumo de productos que no afecten su salud y cubran sus exigencias de calidad. Por esta razón, la industria heladera tiene como objetivo diseñar nuevas formulaciones con excelentes propiedades texturales y sensoriales. Compuestos como inulina y fructanos de agave han sido estudiados debido a los grandes beneficios que estos tienen como prebióticos naturales, fibra dietética y sus funciones tecnológicas (estabilizantes, gelificantes, endulzantes, etc.) (Apolinário y col., 2014; Espinosa y Urias, 2012). Sin embargo, no hay suficiente información acerca de cómo su uso en helados afecta propiedades sensoriales, térmicas y de textura.

Los ingredientes y las condiciones de procesamiento, especialmente durante los períodos de fluctuaciones de temperatura son importantes en la fabricación de helados, ya que determinan su percepción sensorial y de textura. Las propiedades sensoriales (sabor, textura y apariencia) son características de los alimentos detectadas por los sentidos de la vista, olfato, gusto, tacto y audición. Para los helados, la percepción de sabores puede verse afectada porque se perciben menos intensos a bajas temperaturas. La textura es otro parámetro sensorial importante que generalmente es detectado por medio de los sentidos del tacto. El sentido táctil es el método principal para detectar la textura, pero también se utilizan la cinética (sentido del movimiento y la posición) y, a veces, la vista (tasa de flujo) y el sonido (asociado con texturas nítidas, crujientes y agrietadas) (Costa y col., 2015). Para evaluar la textura la industria alimenticia ha utilizado el perfil descriptivo para proporcionar una representación cualitativa y cuantitativa de aspectos que son percibidos por el ser humano, lo que permite medir la reacción sensorial a los estímulos a partir del uso de un producto. Todos los análisis descriptivos implican la detección (discriminación) y la descripción de aspectos sensoriales tanto cualitativos como cuantitativos de un producto que son relevantes para los consumidores, pero que utilizan evaluadores capacitados para la evaluación real (Torres y col., 2017).

Las propiedades térmicas de los helados son importantes para determinar los tiempos de congelación y simular las variaciones del campo de temperaturas durante la congelación, así como los períodos de almacenamiento. Lograr un control de las propiedades térmicas mejora la calidad y la estabilidad de los helados. Estas propiedades se han estudiado para simular el proceso de congelación, que tiene una gran importancia en el diseño de equipos y para el control de sus costos operativos (Cogné y col., 2003).

Por otro lado, el control instrumental de la textura y la calidad del helado se realiza comúnmente mediante el análisis de varias propiedades, como es la viscosidad de la base (mezcla antes de congelar), mediciones de derretimiento, de compresión y penetración. Estas mediciones las hemos analizado previamente en helados reducidos en grasa y helados reducidos en grasa y azúcar (Pintor y Totosaus, 2012; Pintor y col., 2013; 2017).

La reducción de grasa y azúcar generalmente provoca cambios en la microestructura que se ve reflejado tanto sensorial, térmica y texturalmente. Por lo tanto, el uso de compuestos que imiten la funcionalidad de compuestos tan importantes como grasa y azúcar son de vital importancia en la formulación de helados.

1.2 Justificación

Actualmente el desarrollo de productos alimenticios se ve en la necesidad de producir nuevos alimentos con características deseables y aportes calóricos moderados. Las líneas actuales de investigación ya no sólo se centran en el consumo de energía y nutrientes (proteínas, grasas, hidratos de carbono, vitaminas) sino también en el consumo de otros compuestos que sirven como protectores a nuevas enfermedades, como es el caso de las fibras y antioxidantes presentes principalmente en los vegetales.

El helado es considerado como un producto nutritivo que proporciona gran cantidad de energía debido a su alto contenido de proteínas y grasas. Sin embargo, una limitante de su consumo es la gran contribución calórica que éste contiene. El reto para los fabricantes de helados reducidos en grasas y carbohidratos es remplazar estos compuestos por otros que mimeticen sus propiedades sin alterar sus características sensoriales. El uso de compuestos como la inulina o fructanos de agave para reducir el contenido calórico en helados, además de inferir positivamente en las propiedades fisicoquímicas tiene un efecto positivo al actuar como prebiótico, lo que provoca efectos benéficos para el consumidor (Akin y col, 2007; Akalin y col, 2008; Celso y col., 2018; El-Nagar y col, 2002; Karaca y col, 2009; Soukoulis y col, 2009; Mehdi y col., 2016). Además se ha usado como prebiótico en helados con lactobacilos (Balthazar y col., 2018).

La interacción de estos ingredientes afecta la funcionalidad del sistema lácteo congelado como el helado, que además de ser una espuma es una emulsión estabilizada por la proteína del sistema, donde la cantidad de grasa y azúcar son importantes para mantener el balance estructural del sistema (esto es, la cristalización del agua –modificada por el contenido de sólidos– para crear la estructura característica), lo cual se verá reflejado tanto en el rendimiento como en la textura del producto. De ahí la importancia que tiene el reemplazar la grasa y el azúcar, por ingredientes con las propiedades funcionales adecuadas, a fin de compensar textural y sensorialmente al helado. Además, el uso de estos compuestos brinda al consumidor nuevas opciones de helados con buenas propiedades nutricionales.

2. Objetivos e Hipótesis

2.1 Objetivo general

Estudiar las propiedades fisicoquímicas, térmicas y sensoriales en helados reducidos en grasa y azúcar para ver el efecto de inulina y fructanos como remplazo.

2.2 Objetivos específicos

- Estudiar la reducción simultánea del contenido de grasa y azúcar, empleando inulina de achicoria para compensar estos ingredientes, mediante la metodología de superficie de respuesta. Utilizando como variables de respuesta viscosidad, overrun, propiedades de derretimiento (tiempo de primera gota y velocidad de fusión) y de textura (fuerza de compresión y penetración).

- Evaluar el efecto de la reducción de grasa y azúcar, empleando fructanos de agave como remplazo en dos tipos de helados, reducidos en grasa (LF) y reducidos en grasa y azúcar (LFS), sobre las propiedades térmicas: agua congelada y no congelada, temperatura de transición vítrea y propiedades de derretimiento. Asimismo, se buscó explorar posibles interacciones moleculares de los fructanos de agave con la matriz del helado por medio de espectrometría infrarroja.
- Evaluar el efecto de fructanos de agave como remplazo de grasa y azúcar sobre las propiedades sensoriales, así como su correlación con las propiedades térmicas y de textura.

- Caracterizar sensorialmente muestras de helados comerciales y prototipos (con fructanos de agave) por medio de un análisis descriptivo, así como evaluar el efecto que tienen los fructanos de agave sobre las propiedades sensoriales dinámicas de helados reducidos en grasa y azúcar, usando las metodologías TDS y TCATA.

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2.3 Hipótesis

Cambios en la concentración de ingredientes clave en la formulación de helados, como son la fracción de grasa y azúcar, se verán compensadas tanto funcional como sensorialmente por la incorporación de inulina de achicoria y fructanos de agave. Los fructanos de agave modificarán positivamente las propiedades de textura, provocando helados más suaves y con tiempos más largos de derretimientos, además de modificar positivamente las propiedades térmicas producto de la retención de agua libre.

3. Revisión bibliográfica

3.1 ¿Qué es el helado?

El helado es un producto ampliamente reconocido a nivel mundial. El término "helado" en cierto sentido, cubre un amplio rango de diferentes tipos de postres congelado, como son:

- Helado de leche: es una mezcla aireada y congelada de ingredientes lácteos, azúcar y sabor.
- Helado no lácteo: hecho con proteína de leche u otra proteína y grasa vegetal.
- Gelato: es un helado estilo italiano a base de natilla el cual contiene yemas de huevo.
- Yogurt congelado: el cual puede contener ácido láctico o simplemente sabor a yogurt

- Congelado de leche: similar al helado de leche, pero sin airear y con menos grasa de leche.
- Sorbete: es a base de agua y fruta con textura aireada, pero sin contenido de grasa o leche.
- Sherbet: similar al sorbete, pero con contenido de leche o crema.
- Congelado de agua o nieve: mezcla dulce y congelada la cual puede contener colorantes, saborizantes y frutas, como una “paleta de hielo”.

La definición legal del helado varía dependiendo el país. En Reino unido el helado es definido como un producto congelado que contiene como mínimo el 5% de grasa y 2.5 de proteína de leche, sólidos de leche y azúcar (o edulcorantes). En EU el helado por lo menos debe contener el 10% de grasa de leche y 20% de sólidos totales lácteos y debería pesar por lo menos 0.54 kg por litro. De acuerdo con la calidad y cantidad de los ingredientes, así como al contenido de grasa láctea y aire incorporado al helado, éste puede ser caracterizado como premium, standard o económico.

En su mayoría la gente se encuentra familiarizada con la apariencia, el sabor y la textura del helado sin embargo pocos saben el porqué de los ingredientes y de su complejidad en cuanto al proceso. Lo cierto es que el helado es un producto extremadamente complejo, de hecho, ha sido considerado el producto coloidal más complejo de todos. La ciencia del helado consiste en el entendimiento de sus ingredientes, proceso, microestructura, textura, así como la conjunción de ellos. Esto involucra varias disciplinas científicas como la fisicoquímica, ciencia de alimentos, ciencia de coloides, ingeniería química, microscopía, ciencia de los materiales y ciencia del consumidor. Los ingredientes, así como el proceso crean la microestructura (Figura 1). Esta consta de cristales de hielo, burbujas de aire, y gotas de grasa que van desde 1µm hasta 0.1 mm, una solución viscosa de azúcar, polisacáridos y proteínas de leche conocida como matriz o suero. Marshall y col. (2009) y Clarke (2004) entre otros autores, sugieren que el helado es una mezcla aireada y congelada que se mantiene por debajo de los -20°C de forma homogénea y consta de tres principales componentes estructurales, estos son: células de aire, cristales de hielo y glóbulos de grasa, que se encuentran inmersos en una fase líquida de alta viscosidad con azúcares, proteínas de leche y agua no congelada, denominada suero.

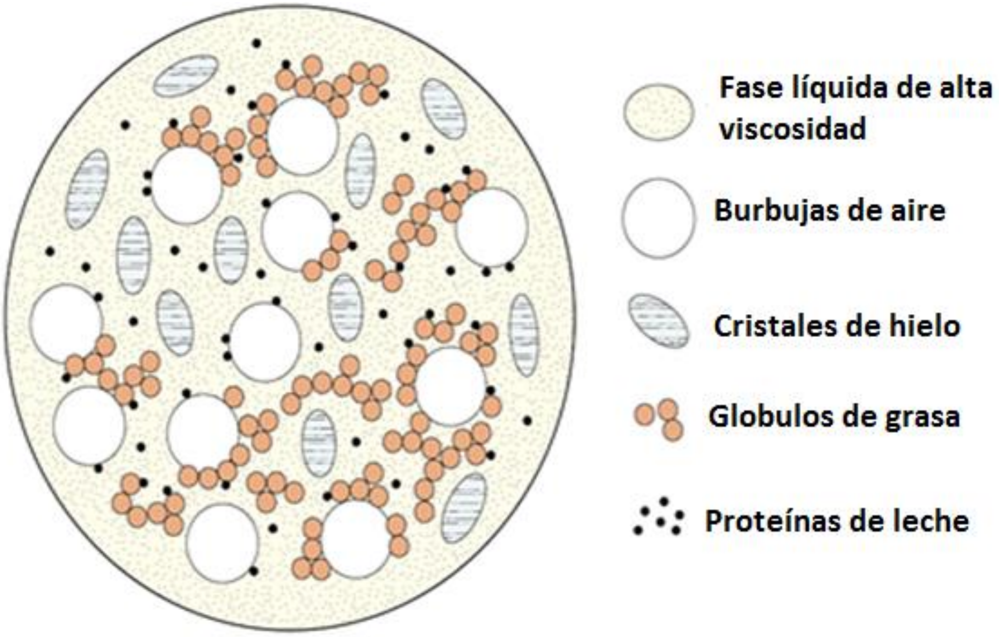


Figura 1. Diagrama esquemático de la microestructura del helado.

3.2 Historia del helado

La historia y evolución del helado está llena de mitos e historias bien fundamentadas que datan de miles de años atrás (Figura 2). Para conocer el origen del helado debemos remontarnos por lo menos seis mil años atrás. Los chinos ya usaban el hielo para conservar alimentos, así como unos tipos congeladas con azúcar que vendían en las calles de Pekín, por lo tanto, podemos decir que ellos son los originarios de tan rico postre.

Este conocido producto se expandió por diversas culturas del viejo mundo. Ya en tiempo del antiguo Egipto, el helado se llevaba a la mesa del faraón y se servía en los banquetes en copas de plata, era una especie de granizada de frutas (Figura 3). En cambio, los persas lo servían en la mesa de los potentados, y de ellos aprendieron los griegos, que se aficionaron a esta golosina cuando Alejandro Magno la probó por primera vez, en el siglo IV a.C. El helado era muy popular en la antigua Roma**,** no sólo entre el pueblo sino también entre las clases elevadas. A Nerón le encantaba, pero como hombre cauto mandaba hervir el agua antes de introducirla en la olla donde luego se elaboraba (Clarke, 2004).

Otra típica historia empieza con el emperador Romano Nero (37-68 d.C) el cual se dice descubrió el principio del helado cuando comía las frutas congeladas que rodaban de las montañas. Otra conocida historia es la de los caballos Mongoles, en los cuales se llevaba crema en intestinos de animales para aguantar las largas jornadas que se llevaban en el desierto de Gobi durante el invierno. La fricción del movimiento del caballo hacía que la crema se congelara con las bajas temperaturas.

La expansión del imperio Mongol llevó esta idea hasta China, de donde Marco Polo supuestamente compró la idea para Italia en el año 1925. Por otro lado, se ha reclamado que el helado fue introducido a Francia por los italianos cuando 14 años atrás, Catherine de Medici contrajo matrimonio con el Duque d´Orléans en 1533 (Clarke, 2004).

4000 a.C.
Aparece el primer helado. Era una pasta de arroz hervido.

400 a.C.
En Persia se sirve un plato enfriado hecho de agua de rosas. Se mezclaba con azafrán, frutas y otros sabores.

320 a.C.
Alejandro Magno hace enterrar en la nieve cantaros llenos de fruta y miel para consumirlos como postres.

60
Nerón enfriaba sus jugos de fruta y sus vinos con hielo o nieve traídos de las montañas por sus esclavos.

1100
Los cocineros árabes de los califas de Bagdad incorporaron jugos de fruta a la preparación. Se llamaban "Sharbets".

1553
Catalina de Médicis se casa con Enrique II de Francia, su cocinero llevó primitivas recetas de helados italianos a la corte francesa.

1660
Francesco Procopio crea el "Café Procope" e inventa una máquina que homogenizaba las frutas, el azúcar y el hielo.

1920
El estadounidense Christian Nelson crea la barra helada.

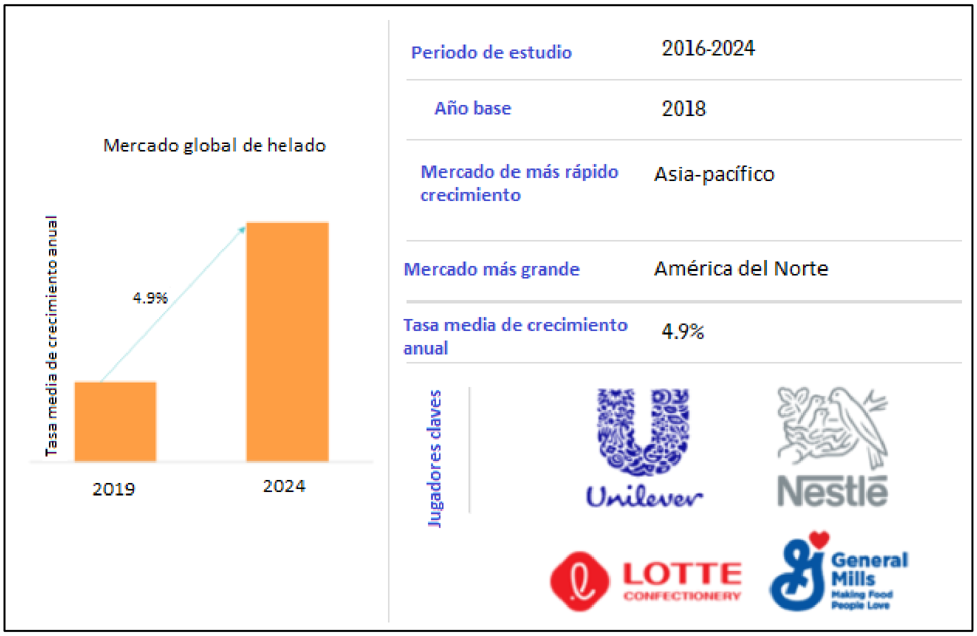
Figura 2. Evolución cronológica del helado (Clarke, 2004).

Figura 3. Inscripciones relacionadas con el uso y /o consumo de “helado” en el antigua Egipto por faraones (Clarke, 2004).

3.3 Mercado global del helado

El helado se hace y se come en todos los países del mundo. La producción mundial de helados y productos congelados fue de 16.3 billones de litros en el 2017, un promedio de 2.4 por persona (Figura 4). Los mayores productores son Asia y América del Norte con 4.9 y 4.3 billones de litros, respectivamente. Países como Nueva Zelanda y Estados unidos tienen el mayor consumo anual per cápita (26 y 22 litros per cápita) en comparación con México que es de 3 litros per cápita. Un estudio generado por Mordor Intelligence en el 2018 (Figura 5), reveló que del 2019 al 2024 el mercado global del helado incrementará hasta un 4.9%, siendo Norte América el mayor productor de éste. En México, helados, congeladas y paletas son productos adquiridos en 7 de cada 10 familias, en especial donde hay niños. En temporada de calor su consumo incrementa hasta el 48% (Fearon, 2018).

Figura 4. Tamaño del mercado del helado a nivel mundial en el 2018.



Periodo de estudio	2016-2024
Año base	2018
Mercado de más rápido crecimiento	Asia-pacífico
Mercado más grande	América del Norte
Tasa media de crecimiento anual	4.9%

Figura 5. Datos estadísticos del mercado del helado a nivel mundial.

3.4 Consideraciones fisicoquímicas del helado

3.4.1 Dispersiones coloidales

Un helado común contiene cerca del 30% de hielo, 50% aire, 5% grasa y 15% matriz o suero (solución de azúcar) por volumen. Por lo tanto, contiene todos los estados de la materia: después del batido y la congelación, hielo y grasa (sólido), antes de congelar solución de azúcar (líquido) y después del batido, burbujas de aire (gas). Los sólidos y el gas están en forma de pequeñas partículas, los cristales de hielo, glóbulos de grasa, y burbujas de aire están en una fase continua líquida, la matriz. Para entender la creación de la microestructura durante la manufactura del helado es importante conocer algunos conceptos fisicoquímicos de los coloides.

Las dispersiones coloidales consisten en pequeñas partículas de una fase (sólido, líquido o gas) en una fase continua. Estas dispersiones tienen un área superficial muy grande para su volumen. Por lo tanto, las propiedades de la superficie tienen una gran influencia sobre las propiedades como un todo. El helado es simultáneamente una emulsión (glóbulos de grasa), un sol (cristales de hielo) y una espuma (burbujas de aire) y también tiene otros coloides en forma de micelas de caseína, otras proteínas y polisacáridos en la matriz (Soukoulis y col., 2009).

3.4.1.1 Emulsión

Es un sistema constituido por dos fases inmiscibles (que no se mezclan), una de las cuales se dispersa a través de la otra en forma de gotas muy pequeñas. En la mayoría de las emulsiones, una de las fases es acuosa y la otra un aceite. Las emulsiones con el aceite como fase dispersa se conocen como emulsiones de aceite en agua (oil-in-water, o/w) y las emulsiones con agua como fase dispersa se conocen como emulsiones de agua en aceite (water-in-oil, w/o). Por ejemplo, el helado es una emulsión aceite en agua (o/w). El tipo de emulsión depende del balance entre las propiedades hidrófilas e hidrófobas del agente emulsificante. Generalmente se suele cumplir la regla de Bancroft: la fase continua es aquella la cual solubiliza al agente emulsificante. La naturaleza anfótera de los agentes tensioactivos puede ser expresado en términos de una escala empírica que comúnmente se denomina el balance HLB (balance hidrófilo-lipófilo).

Una propiedad de los líquidos es la capacidad de formar interfaces debido a las capas de moléculas cuyas fuerzas de cohesión las mantienen unidas. Dichas fuerzas no son iguales en la superficie y en el interior del líquido, aunque en promedio terminan anulándose. Como las moléculas de la superficie tienen más energía el sistema tiende a minimizar el total de energía a partir de una reducción de las moléculas superficiales; de este modo el área superficial disminuye. En estos sistemas existe energía libre entre la interfase de los dos líquidos inmiscibles debido al desbalance de las fuerzas cohesivas de los dos materiales. Por lo tanto, podemos decir que las emulsiones son sistemas termodinámicamente inestables, esta inestabilidad se debe al incremento del área de las gotas de grasa que producen un aumento en la entalpía libre de Gibbs. Esta desestabilización se debe a los cambios que ocurren a nivel microscópico y son explicados por una teoría llamada DLVO (Derjaguin, Landau, Verwey y Overbeek) y describen cualitativamente las interacciones entre las gotas. Esta teoría asume que la estabilidad de la emulsión es debida principalmente a las interacciones de largo alcance que ocurren entre las gotas, considerando dos tipos de fuerzas: las fuerzas de Van der Waals (V_A) que son atractivas y de largo alcance las fuerzas electrostáticas (V_R) que son repulsivas debido a las cargas que se encuentran en la superficie de las gotas (tanto agentes tensoactivos como específicamente por iones que se encuentran adsorbidos), creando una energía de repulsión. El potencial total de interacción $V= V_{A} + V_{R,}$ está en función de la distancia (d) que existe entre las gotas. La combinación de ambas fuerzas es llamada energía neta de interacción. El valor neto se representa arriba si es repulsivo o abajo si es de atracción, formando la curva de interacción. Si existe una zona repulsiva, entonces el punto de máxima energía de repulsión se llama barrera de energía, la altura de esta barrera indica cuan estable es el sistema (Figura 6) (Méndez y Goff, 2012).

Para aglomerar 2 partículas que van a chocar, estas deben tener suficiente energía cinética debido a sus volumen y masa, como para pasar dicha barrera. Si esta barrera desaparece, entonces la interacción neta es totalmente atractiva y consecuentemente las partículas se aglomeran, llegando a un nivel de mínima energía que involucra la separación de fases (Coubet y col., 2011).

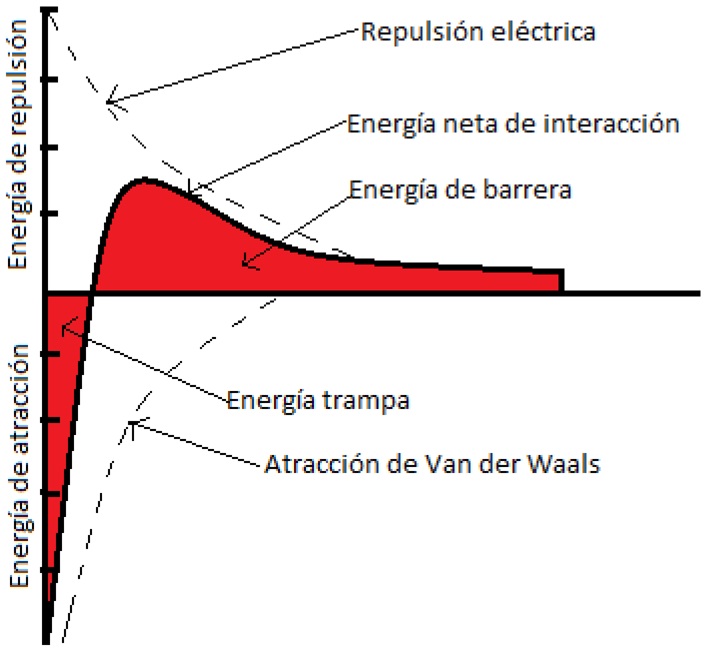


Figura 6. Diagrama de la estabilización de una emulsión debido a interacciones de atracción y repulsión.

La estabilidad física de las emulsiones puede romperse debido a cuatro tipos de fenómenos:

- Sedimentación/ cremado: donde las partículas se concentran en la superficie o en el fondo, dependiendo de la densidad relativa de las dos fases de la mezcla. Éste efecto es originado como consecuencia de las densidades de ambas fases.
- Agregación: Es originada como consecuencia de las fuerzas de atracción entre las películas formando agregados de partículas.
- Coalescencia o ruptura de la emulsión: Es la fusión de gotas para crear unas gotas más grandes, formando una capa del líquido. Se origina como

consecuencia de la tensión interfacial y causa un cambio real en el tamaño de las gotas.

- Engrosamiento de gotas (Ostwald ripening). Se debe al crecimiento de las gotas más grandes a costa de las más pequeñas hasta que éstas últimas prácticamente desaparecen. Este proceso ocurre a una velocidad que es función de la solubilidad de la fase dispersa en la fase continua y se debe a que la presión interna de las gotas (presión de Laplace) es mayor en las gotas más pequeñas.

Conforme transcurre el tiempo la emulsión tiende a separarse, sin embargo, este efecto puede ser contrarrestado con la adición de emulsificantes y estabilizantes. Los emulsificantes son agentes superficiales activos que facilitan la mezcla de dos o más sustancias líquidas que se separarían en sus partes componentes en condiciones normales, en los cuales sus moleculas poseen una parte hidofílica y otra lipofílica o hidrofóbica (generalmente una parte polar y una no polar). Su esctuctura se puede ver en la Figura 7, donde la cabeza polar (hidrofílica) puede llevar una carga positiva o negativa y es esta parte la que define al agente tensoactivo como un agente catiónico o aniónico respectivamente, mientras que la cola no polar (lipofílica) generalmente suele ser una cadena longitudinal de hidrocarbonos. En helados, los emulsificantes ocupan la superficie limitante entre ambas fases, haciendo disminuir la tensión interfacial y desestabilizando la grasa, es decir, ayudan a que se produzca una cierta coalescencia y agregación de las gotas de grasa para que pueda ser formada la red que estabiliza las burbujas de aire y por lo tanto la estructura del helado. Los tipos de emulsificantes mas usados son los monogliceridos insaturados que por su menor número de fusión logran una mayor desestabilización de la emulsión y una mayor resistencia a la fusión que los saturados. La estabilidad de la emulsion depende también de otros factores aparte del emulsificantes, como es el tamaño de los globulos de la fase dispersa, la diferencia de densidades entre ambas partes, la viscosidad de la fase continua y de la emulsion, las fuerzas de interfase que actúan en la superficie de los glóbulos, la naturaleza eficacia y cantidad de emulsificante y estabilizante y finalmente las condiciones de almacenamiento (Méndez y Goff, 2012).

Los estabilizantes son polimeros absorbentes del agua que tiene la capacidad de reducir la cantidad de agua libre, absorbiendo parte de las moleculas de agua por enlaces de hidrógeno. No toda el agua es absorbida porque el proceso es suplementado por una inmobilización del agua y se forma una red tridimiencional que reduce la movilidad del agua que queda. Esta absorción/inmovilización del agua aumenta la viscosidad y en algunos casos se forma una estructura de gel en la soción (Soukoulis y col., 2010).

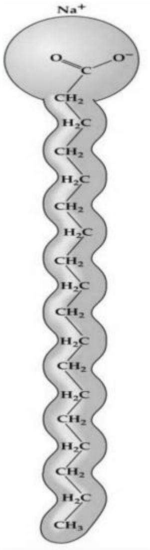


Figura 7. Estructura de un emulsificante donde se muestra su cabeza polar y su cola no polar.

3.4.1.2 Soles

El helado también es considerado un sol, que son dispersiones de partículas sólidas en una fase líquida continua (cristales de hielo en el helado), donde la fase sólida es significativamente más densa que la fase líquida. En el helado los movimientos brownianos son el movimiento aleatorio de partículas coloidales (cristales de hielo) suspendidas en un líquido o gas, causado por colisiones con las moléculas del medio circundante (fase continua). Las partículas en soluciones y coloides están en constante movimiento. Sin embargo, las partículas coloidales son lo suficientemente grandes como para ser observadas y lo suficientemente pequeñas como para verse afectadas por las colisiones moleculares aleatorias. Las partículas coloidales se resisten a asentarse rápidamente en el fondo de un vaso debido al movimiento browniano (Clarke, 2004; Méndez y Goff, 2012).

3.4.1.3 Espumas

Una espuma es una dispersión de burbujas de aire en un volumen relativamente pequeño de una fase continua liquida. Las espumas liquidas consisten de burbujas de gas separadas por una pequeña capa liquida la cual contiene moléculas con superficie activa (emulsificantes, proteínas) que son absorbidas en el gas o en la interface liquida que ayudan a estabilizar el helado (Clarke, 2004).

3.5 Función de los componentes del helado

El helado es un producto muy variable, debido a que su formulación y proceso de elaboración puede cambiar dependiendo el país o el fabricante. Aunque pueden ser empleados muchos ingredientes, el uso de compuestos básicos como sólidos de leche, grasa, estabilizantes y emulsificantes (base peso/peso), son indispensables (NOM-036-SSA1 1993). Cada uno de estos compuestos juega un rol especial sobre la formación de la estructura.

Agua: en helados es conocida como la fase continua y es el componente en donde todos los solutos se encuentran dispersos.

Aire: es otro ingrediente básico que conforma la estructura del helado. Éste tiene que estar libre de aromas y microorganismos. Cuanto más alto es el contenido de sólidos en el helado, más cantidad de aire es incorporado. En helados, el porcentaje de overrun es la manera de medir el aire que se introdujo durante el batido.

Grasa: la grasa que se incorpora en el helado puede ser de origen lácteo, vegetal o ambas. La grasa juega un papel esencial en el helado: disminuye la tendencia al derretimiento, tiene un efecto estabilizante, promueve la incorporación y dispersión de aire, incrementa la viscosidad, imparte el aroma y favorece la formación de cristales de hielo (Bolliger y col., 2000; Chung y col., 2003; Clarke, 2004; Granger y col., 2005; Méndez y col., 2012). Su efecto estabilizante se debe a la formación de agregados de glóbulos grasos que forman una red tridimensional que atrapan las burbujas de aire, estos agregados son el resultado de la coalescencia parcial que ocurre durante el batido. Durante la agitación de los glóbulos de grasa (que contienen gran parte de los triglicéridos cristalizados), se rompe la película protectora y al aproximarse quedan enganchados por el contacto grasa/grasa, es la grasa cristalizada la que impide que la coalescencia sea completa, formándose agregados de forma irregular que se unen entre sí, constituyendo una red continua en la matriz del producto (Chung y col, 2003). La capacidad de la grasa de promover y mantener la dispersión de aire en el helado es debido a que la grasa se coloca en la superficie de las burbujas de aire proporcionándoles una fina capa que las estabiliza. Este efecto impide que durante el almacenamiento las burbujas de aire interactúen entre sí y escapen del producto. Para que esto se logre es necesario que exista suficiente grasa que cubra toda la superficie de las burbujas.

Carbohidratos: este compuesto es adicionado usualmente entre 12-17 p/p del volumen total de la mezcla. Proporcionan el sabor dulce, influyen sobre el punto de congelación, incrementan la viscosidad mejorando la textura y palatabilidad del helado. Los azúcares también disminuyen la dureza de los helados. En particular azúcares y estabilizantes son compuestos importantes que determinan el tamaño de los cristales de hielo (Hagiwara, 1996). Los azúcares usados más comúnmente son: la sacarosa, fructosa, lactosa, jarabe de la glucosa, azúcar invertida y los alcoholes de azúcar glicerol y sorbitol.

Sólidos lácteos no grasos: Estos compuestos son usados en el helado de 10-14 p/p y son conformados principalmente por proteínas lácteas y sales minerales, que tienen por objeto estabilizar y aumentar la viscosidad de la mezcla base para helado. Las proteínas interactúan con el agua libre, dando al helado una textura suave y con buena consistencia. En conjunto con emulsificantes y estabilizantes determinan las propiedades reológicas del producto (Clarke, 2004).

La composición aproximada de la leche en polvo es 36% de proteína láctea (caseína y productos solubles), 56% de lactosa y 8% de materiales minerales. La proteína láctea en helados actúa como emulsionante durante la homogenización de la mezcla y como agente tensoactivo durante el proceso de congelación. Es necesario que las proteínas lácticas después de la homogenización se coloquen sobre la superficie de los glóbulos de grasa para evitar una coalescencia excesiva. Esta función además de las proteínas del suero también pueden hacerlo las caseínas. La lactosa junto con las matrerías minerales y azucares disminuyen el punto de congelación de la fase acuosa. El único inconveniente de la lactosa en helados es que es poco soluble y que si hay fluctuaciones de temperatura y almacenamientos prolongados esta recristaliza formando texturas arenosas (Alvarez, 2005).

Estabilizantes: son un grupo de ingredientes (usualmente polisacáridos) usados en la elaboración de helados en concentraciones mínimas (<1%). El objetivo principal del uso de estos compuestos es provocar suavidad, cuerpo y textura en los helados, retardando o reduciendo el crecimiento de cristales de hielo y lactosa durante el almacenamiento, especialmente durante los periodos de fluctuación, impartiendo uniformidad y resistencia al derretimiento.

También incrementan la viscosidad de la mezcla, promueven la incorporación de aire y estabiliza al sistema contra la separación de fases, lo cual se ve reflejado en caracteristicas sensoriales deseables como suavidad y largos periodos de derretimiento (Akesowan, 2008; Hernández y col., 2011; Philip y Laaman, 2011). El trabajo más importante de los estabilizantes es ligar agua libre contenida en la fase liquida, impartiendo textura al helado. Se sabe que las carrageninas son hidrocoloides que son usados por su habilidad de interactuar con proteínas para formar geles o suspensiones viscosas. Otros compuestos como goma guar, goma de algarrobo, goma xantana, goma tara, goma de celulosa y celulosa microcristalina son utilizadas para reducir la movilidad de agua (Clarke, 2004).

Inulina: es un carbohidrato no digerible que se encuentra presente en vegetales, frutas y cereales, aunque actualmente a nivel industrial se extrae de la raíz de achicoria (*Cichorium intybus*). Está compuesto principalmente por cadenas lineales de fructosa. El uso de la inulina más extensamente estudiada es su comportamiento como prebiótico, actuando como un estimulante de crecimiento bacteriano en el colon, con la consecuente disminución de otras especies que pueden ser dañinas. El aporte de fibra dietética, bajo valor calórico (1.5 kcal/g), su capacidad de espesar, emulsificar, gelificar, humectar, sustituir azucares y grasas, son algunas de sus propiedades (Madrigal y Sangronis, 2007).

Fructanos de agave

Este compuesto consiste en una estructura ramificada muy compleja de fructooligosacáridos unidos por enlaces β (2-1) y β (2-6) con moléculas de glucosa (Espinosa y Urias, 2012). En una determinación de la distribución de fructanos de agave por Cromatografía de Exclusión se determinó que el grado de polimerización (DP) fue de 17.1±0.2, teniendo una proporción de fructanos (DP > 1) de 72.5 g/100g y FOS Fructooligosacáridos) (DP 3-10) de 27.4g/100g con contenido residual de sucrosa (0.48g/100g), glucosa (1.88g/100g) y fructosa (4.94g/100g).

En comparación con la inulina de achicoria (fructanos con estructura lineal), los fructanos de agave han mostrado tener una mayor capacidad de absorción de agua. Los fructanos de agave han sido usados en yogurts reducidos en grasa con el propósito de mejorar positivamente su microestructura, propiedades reológicas y sensoriales. En trabajos anteriores hemos estudiamos el efecto de inulina de achicoria en helados bajos en grasa butírica (Pintor y col., 2013), así como el efecto de fructanos de agave en helados bajos en grasa y azúcar (Pintor y col., 2017).

3.6 Manufactura del helado

El proceso de manufactura del helado consta de una serie de pasos que se presentan a continuación (Figura 8).

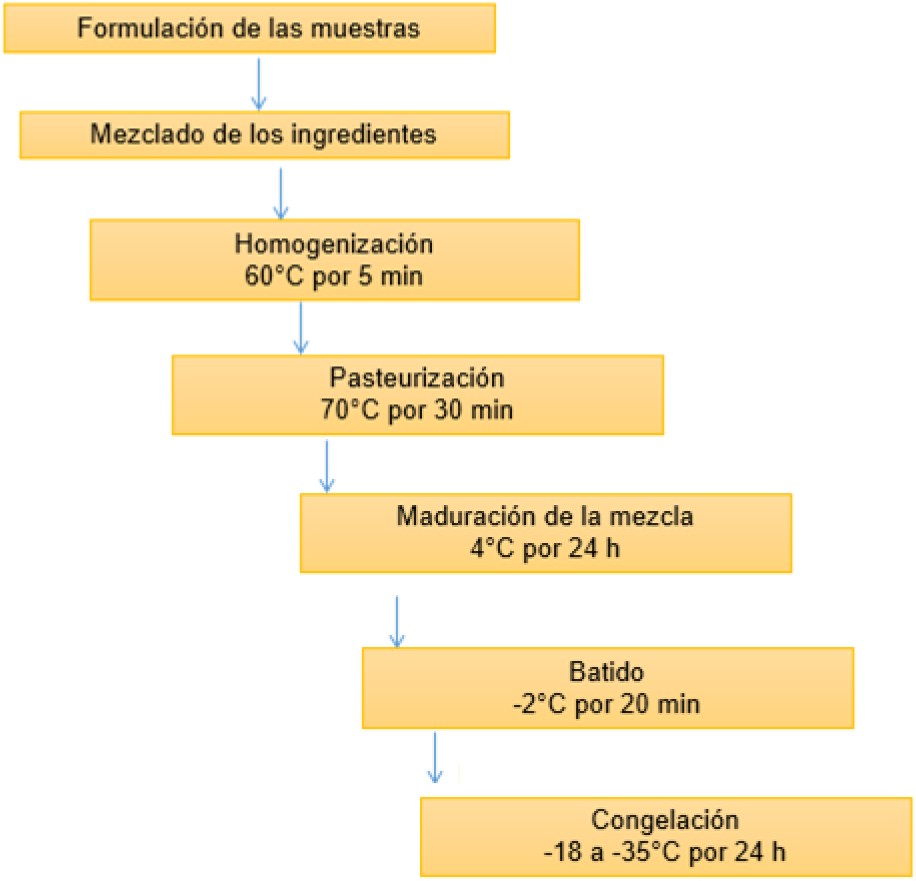


Figura 8. Proceso de elaboración del helado

El primer paso es la formulación y mezclado de los ingredientes, el cual tiene por objeto dispersar e hidratar los ingredientes que conforman el helado (leche en polvo, suero de leche, grasa butírica, grasa vegetal, mezcla de estabilizantes y azúcar). Estos ingredientes son dosificados en cantidades exactas y agregados en un orden especial. Primero son adicionados los componentes líquidos (agua), seguido de los componentes secos (leche en polvo, suero de leche, grasa, emulsificantes, estabilizantes y azúcar), en el caso de los estabilizantes es necesario hacer un pre-mezclado a temperaturas donde los polímeros son hidratados con el fin de evitar la formación de grumos en la mezcla. Por ejemplo, goma de algarrobo, iota y lambda carragenina se hidratan bien a temperaturas superiores a los 75°C, por el contrario, carboximetilcelulosa se hidrata a 24°C (Clarke, 2004).

Homogenización y Pasteurización

La homogenización es el proceso responsable de la formación de la emulsión a través de movimientos mecánicos y temperaturas que tienen como objeto la buena distribución de los compuestos, rompiendo las gotas de grasa con el fin de que choquen y se fusionen, ampliando más la superficie de contacto y por consiguiente logrando una emulsión estable que absorbe cualquier tipo de molécula anfifílica que se encuentre en el medio (principalmente proteínas, lipoproteínas, caseínas y emulsificantes) (Figura 9). Por ejemplo, en la leche con un 3.5% de grasa, la homogenización reduce el diámetro del glóbulo de grasa de 3.3 a 0.4 µm, incrementa el área de superficie de 0.08 a 0.75 m^2/ ml y aumenta el número de glóbulos de 0.015 a 12 μm^{-3} (Goff, 1997).

Moléculas como proteínas, emulsificantes y estabilizantes cambian su conformación durante la homogenización. Las proteínas son uno de los compuestos más sensibles ante el proceso, debido a que éstas, a temperaturas cercanas a los 80°C tienden a desnaturalizarse (pierden su estructura tridimensional), provocando precipitación y separación de fases. Otros factores que afectan las propiedades de las proteínas en estos sistemas son la presencia de polisacáridos, polipéptidos de bajo peso molecular, fosfolípidos, azucares, pH y fuerzas iónicas (Clarke, 2004).

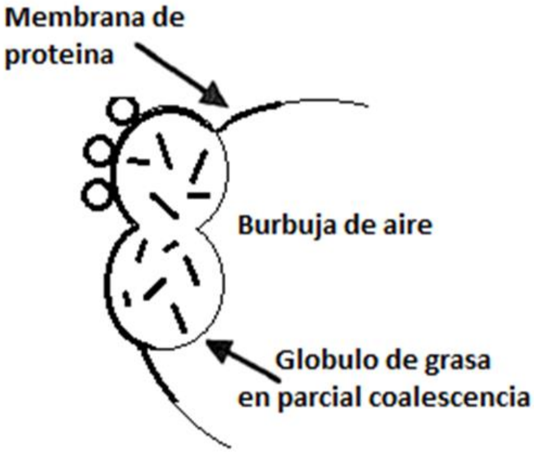


Figura 9. Interacción interfacial entre proteínas, burbujas de aire y glóbulos de grasa en parcial coalescencia

Los fosfolípidos y polipéptidos (emulsionantes) de bajo peso molecular compiten por la adsorción en la superficie de la burbuja de aire, desplazando a las proteínas y por lo tanto desestabilizando la emulsión (Figura 10). A condiciones y cantidades ideales, las proteínas trabajan en conjunto con estabilizantes formando en este caso complejos que aumentan la viscosidad, confiriendo buenas propiedades al helado.

Las proteínas para ejercer su función emulsionante siguen un mecanismo en tres etapas, la primera consiste en su transporte conectivo desde la fase continua a la interfase, la segunda en su adsorción en la interfase y la tercera en una reorganización de su estructura en la interfase, que recibe el nombre de desnaturalización superficial.

Estabilizantes como las carrageninas (concentraciones menores a 0.5 p/p) son ampliamente utilizados en productos lácteos debido a su interacción especifica con las proteínas y capacidad de extender su configuración al interactuar con otros polisacáridos, induciendo zonas de interacción con el agua provocadas por los grupos sulfatos cargados positivamente (Pintor y Totosaus, 2012). En sistemas lácticos donde proteínas y carrageninas están presentes es conocido que aumentan su capacidad de emulsión a pH 6.0 y bajas fuerzas iónicas (0.2 M), lo que se traduce en, que tan capaces son las proteínas de migrar a la interfase agua/aceite, mostrando efecto en su conformación y propiedades. Aunque las proteínas contribuyen en la estabilización de la base para helado, es necesario adicionar compuestos como mono y diglicéridos con la finalidad de mantener la dispersión uniforme. Estos emulsificantes presentan una estructura bipolar, donde se distingue una parte hidrofílica y otra lipofílica que reduce la tensión interfacial entre agua y grasa, lo que resulta en el desplazamiento de las proteínas sobre la superficie del glóbulo de grasa y por lo tanto facilita la interacción entre compuestos.

La pasteurización es el proceso posterior a la homogenización que mantiene el control biológico del helado, destruyendo bacterias patógenas que son adquiridas durante la manipulación de los ingredientes. Los tiempos y temperaturas de pasteurización dependen del tipo de componentes que conforman la mezcla. Una pasteurización lenta reduce el 99% de los microrganismos patógenos a temperaturas de 62-72°C en tiempos de 8-40 minutos. La pasteurización rápida reduce la carga microbiana en un 95.5% a temperaturas de 71-74°C en un tiempo de 40-45 segundos (Goff y Hartel, 2004). En este proceso se terminan de solubilizar proteínas y estabilizantes inmersas en el sistema.

Maduración de la mezcla

Después del enfriamiento es recomendable reposar la mezcla por más de 4 horas con el fin de que se terminen de hidratar las proteínas de leche y estabilizantes. Esto permite el aumento de viscosidad, afectando positivamente la textura del helado y por lo tanto la calidad. A medida que la mezcla se enfría, los mono y diglicéridos comienzan a cristalizar, lo que los hace más hidrofóbicos, de manera que se absorben más fuertemente sobre las gotitas de grasa (Figura 10).

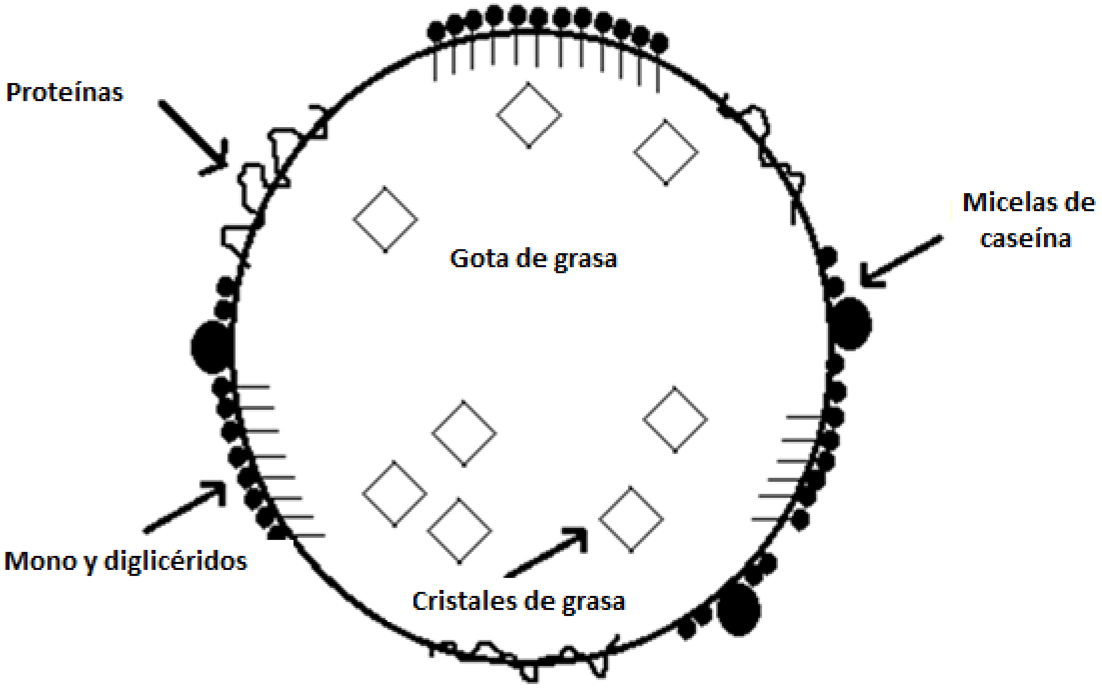


Figura 10. Ubicación de las proteínas de leche y emulsificantes sobre la superficie de una gota de grasa

Estos emulsificantes tienen sus cadenas de ácidos grasos en la fase grasa y sus cabezas polares en la fase continua. El desplazamiento de algunas proteínas a la superficie estabiliza ligeramente la emulsión, pero no lo suficiente para soportar condiciones de cizallamiento. Posterior a esto las gotitas de grasa empiezan a cristalizar tomando a los mono y diglicéridos como puntos de nucleación debido al alto punto de fusión de los triglicéridos. Es importante que esta cristalización no sea total, si no que quede un núcleo de grasa líquida. Estos fenómenos son muy importantes para el siguiente paso que es el batido, debido a que las burbujas de aire dependen de qué tan estable sea la emulsión para no colapsar durante el endurecimiento.

Después de transcurrir el tiempo de maduración es oportuno hacer mediciones de viscosidad, cantidad de grasa, sólidos totales y análisis microbiológicos. En este punto pueden ser adicionados ingredientes que son sensibles al calor como frutas, color y sabor (Clarke, 2004).

Batido

Después del proceso de maduración, la mezcla es aireada, batida y congelada. En este paso la cantidad de aire incorporado depende de la estabilidad e interacción de los componentes, así como de la cantidad y calidad de éstos. Conforme la mezcla se bate, las gotitas de grasa chocan y se fusionan ampliando aún más el contacto superficial. Las proteínas y los emulsificantes proporcionan estabilidad a las burbujas de aire contra la coalescencia.

El aire debe estar firmemente distribuido, de manera que las burbujas no se vean a simple vista, por ello es sugerido que el diámetro sea inferior a 100µm. En la representación esquemática de la estructura de la base para helado (Figura 11), se puede observar a los componentes compitiendo por el espacio en la grasa, estando inmersos en la fase liquida las proteínas y emulsificantes. Después de una buena incorporación de aire, el helado adquiere una consistencia cremosa. La estabilidad de este sistema (aire - cristales de hielo -gotas de grasa - fase líquida) dependerá del grado de incorporación de aire que se introduzca al helado, del tamaño de la celda de aire y, fundamentalmente del espesor de la capa que rodea las burbujas de aire. Esta capa está constituida por la grasa parcialmente desestabilizada, proteínas de la leche, estabilizantes y emulsificantes. Si las burbujas de aire tienen un mayor tamaño habrá una mayor área superficial que será más difícil de cubrir, por lo tanto, la misma será más delgada y las células estarán más predispuestas a deformarse por la acción de los cristales de hielo. Si las burbujas de aire se unen entre sí y se escapan de la matriz, el helado no podrá mantener su forma y colapsará. Manteniendo las burbujas de aire firmemente dispersas se impide que los cristales de hielo estén en contacto entre sí y aumenten su tamaño (Clarke, 2004).

Durante el batido se van formando cristales de hielo con diámetros entre 30-50 µm. Es importante que en esta etapa del proceso se congele la mayor cantidad posible de agua libre, puesto que en la siguiente etapa que es el endurecimiento del helado, los cristales aumentarían de tamaño si existiera aún agua disponible, afectando así la textura del helado.

Uno de los problemas que se observa durante el proceso de batido es que si la base de helado rebaza la cantidad de proteína, la grasa sufrirá una desestabilización, provocando un helado grueso y pesado. Al contrario, si la emulsión es demasiado débil por el exceso de emulsionante o contenga muy poca proteína, las gotas de grasa se fusionarían en grandes gotas, que se sentirían a la hora de comer el helado (Goff, 2002).

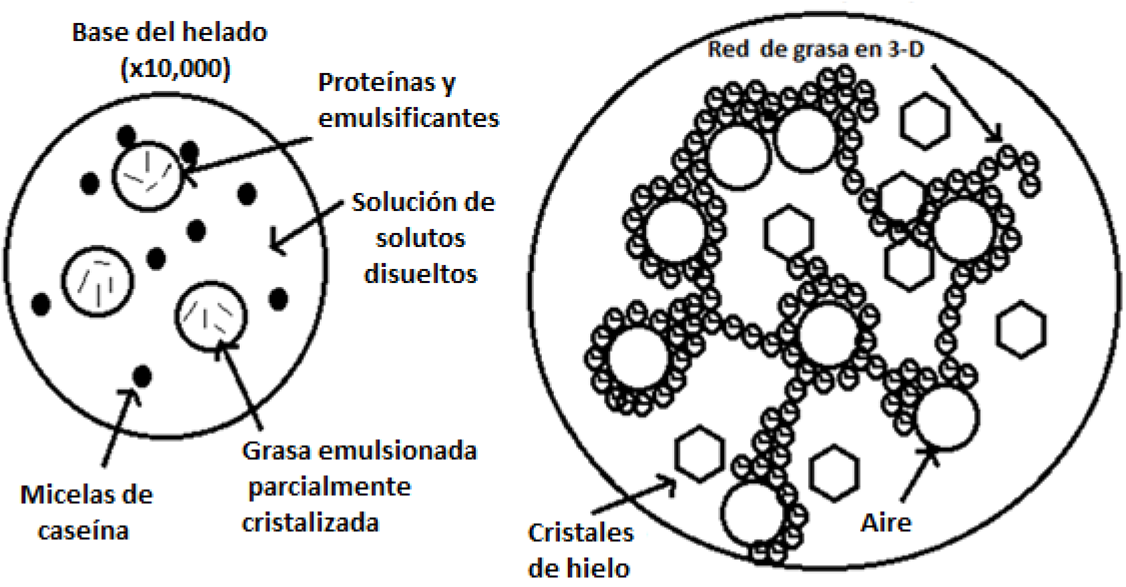


Figura 11. Representación esquemática de la estructura de la mezcla de helado antes (izquierda) y después de congelar (derecha).

Congelación

Uno de los aspectos más importantes de un helado es la textura, está consigue ser óptima cuando la estructura es uniforme, ligera y suave, o cuando las partículas sólidas no se distinguen en la boca. Generalmente se espera que las burbujas de aire midan entre 50-100 µm, los cristales de hielo entre 20-60 µm y los glóbulos de grasa entre 0.2-2.0 µm, además de proteínas, azúcares y estabilizantes que se encuentran en el agua que queda en estado líquido. En este proceso se termina de congelar el agua que queda libre en la matriz del helado. La formación de grandes cristales de hielo es uno de los problemas que causan una textura indeseada. Por ello, después de que la base fue aireada y enfriada se pasa a congeladores de -18 a -30°C con la finalidad de congelar la mezcla rápidamente para evitar la formación de cristales grandes de hielo. En este punto del proceso, los hidrocoloides juegan el papel más importante de todos los compuestos, debido que estos presentan propiedades funcionales que están relacionados estrechamente con la habilidad para retener y conservar grandes cantidades de agua, lo cual influye en las características reológicas y físicas de la mezcla. Afectando positivamente la textura y propiedades de derretimiento del helado. La temperatura a la que es congelada la mezcla afectará también la formación y la velocidad de cristalización. Cuando la mezcla se congela lentamente se produce la nucleación, que es un fenómeno en el cual un cristal pequeño es rodeado por otras moléculas de agua hasta formar cristales de tamaño mayor y por lo tanto una textura defectuosa. Si existe una cantidad apropiada de sólidos totales, la cantidad de agua a congelar se reduce. El contenido de grasa reduce el tamaño de los cristales de hielo y produce un efecto lubricante, lo que provoca una sensación de suavidad en la boca (Goff, 2002).

3.7 Importancia de hacer helados más sanos

El helado es un producto considerado como nutritivo y proporciona una gran cantidad de energía, debido a su contenido alto de proteínas y grasas. Sin embargo, una limitante de su consumo es el gran aporte calórico que proporciona. El contenido de grasas (14-18%) y azúcares (14-17%) es fundamental para la formación de un producto de calidad.

Las grasas de los alimentos además de brindar textura y sabor otorgan grandes beneficios al organismo. Las grasas como todos los nutrientes tienen funciones específicas en el cuerpo, por ejemplo, son fuentes de reserva energética (1g de grasa=9 kcal), son indispensables en la formación de membranas y órganos, son trasportadoras de otras moléculas, etc. Sin embargo, el consumo en exceso de grasas provoca enfermedades cardiovasculares, relacionadas con hígado graso y colesterol alto. La tendencia de consumir alimentos con bajas cantidades de grasa, cada vez es más común entre la población (Underdown y Quail, 2011).

Las grasas saturadas son aquellas que se encuentran sólidas a temperatura ambiente y provocan diversos trastornos en la circulación de la sangre. Sin embargo, son las más empleadas en los alimentos, debido a que provoca una sensación de saciedad y brindan sabores más agradables que las insaturadas. Por esta razón los alimentos más apetitosos son aquellos que contienen más cantidad de grasa.

El azúcar proporciona sabor dulce, influye sobre el punto de congelación, disminuye la dureza, incrementa la viscosidad mejorando la textura y palatabilidad del helado. En particular azúcares y estabilizantes son compuestos importantes que determinan el tamaño de los cristales de hielo. Los azucares más usados son: la sacarosa, fructuosa, lactosa, jarabe de glucosa, azúcar invertida y los alcoholes de azúcar glicerol y sorbitol. Aunque este compuesto es indispensable en la formulación, el alto contenido de estos limita el consumo de helado, principalmente personas con diabetes.

Por lo tanto, reducir grasa y azúcar en helados y sustituirlas por compuestos como inulina o fructanos de agave, sin que afecte su sabor o textura, brinda a los consumidores nuevas alternativas de productos más sanos, además del beneficio que estos compuestos brindan.

3.8 Caracterización del helado

Para el desarrollo de nuevas formulaciones, productos y procesos, por ejemplo, la creación de nuevas texturas o hacer productos mejorados como son los reducidos en grasa manteniendo una textura cremosa, los productores necesitan medir y caracterizar la microestructura y las propiedades del helado. Existen varios análisis que se realizan el helado, los principales son:

1. Visualización y caracterización de la microestructura, por ejemplo, la microscopia óptica o electrónica, para medir cristales (tamaño, número y morfología) de hielo o distribución de tamaño de burbuja.
2. Medidas de respuesta del helado a la deformación, como son propiedades mecánicas o reológicas.
3. Propiedades térmicas, como son la capacidad calorífica, conductividad térmica y temperaturas de fusión.
4. Pruebas microbiológicas para garantizar la inocuidad del producto
5. Mediciones sensoriales, en el cual se usa sistemas sensoriales humanos para evaluar textura, sabor y apariencia.

3.8.1 Importancia de las mediciones de textura

Uno de los aspectos a tener en cuenta para definir la calidad de nuestro helado es que tenga la textura apropiada. Este es un concepto inicialmente visual, y después lo percibimos en la boca, al consumirlo. Se considera que la textura es óptima, cuando: sus componentes proporcionan una estructura cremosa, uniforme, ligera y suave. Esto se refiere a la disposición y dimensión de las partículas sólidas, si son lo suficientemente pequeñas para no ser detectadas en la boca. Las mediciones instrumentales nos ayudan a simular la deformación del helado mientras es consumido. Las pruebas que se hacen al helado antes de su congelación (base de helado), generalmente son pruebas reológicas como es la medición de viscosidad. Después del proceso de congelación es pertinente hacer pruebas de textura (compresión y penetración) y propiedades de derretimiento (velocidad de derretimiento) (Clarke, 2004).

3.8.2 Calorimetría diferencial de barrido

Uno de los objetivos del procesamiento en alimentos es mantenerlos en almacenamiento para prolongar la vida útil del producto. Durante el proceso, existen cambios en los componentes, incluyendo grasas carbohidratos y proteínas. Estos cambios afectan tanto funcional como estructuralmente al producto.

Muchos alimentos son preservados por medio de métodos térmicos (calentamiento, enfriamiento y congelación). La determinación de las propiedades térmicas en alimentos, como el calor específico en función de la temperatura, es esencial para la transferencia de calor y el cálculo del balance de energía. Mediante la calorimetría diferencial de barrido se pueden crear una serie de condiciones para simular, predecir y optimizar procesos. Los datos calorimétricos evalúan la estabilidad termodinámica de varias fases para alimentos y condiciones del proceso.

La calorimetría diferencial de barrido, la cual mide la capacidad calorífica en función de la temperatura, es un análisis térmico que detecta y monitorea las transiciones térmicas y las transiciones de fase en función de la temperatura. Este análisis arroja un termograma donde los picos o puntos de inflexión reflejan la transición térmica que puede ser observada. La dirección de los picos corresponde a la naturaleza de la transición, calor absorbido (endotérmicos) y calor emanado (exotérmicos). El derretimiento de sólidos y la desnaturalización de las proteínas muestran picos endotérmicos, la cristalización de carbohidratos y la agregación de proteínas muestran picos exotérmicos. Tanto las temperaturas de transición (endotérmicas y exotérmicas) como el flujo de calor, son detectados por el calorímetro. Los puntos de inflexión indican la transición vítrea, que es la transición de estado de gomosa a vítrea. Las temperaturas de transición reflejan la estabilidad térmica de la fase o estado a través de la transición. De los datos de calorimetría se pueden obtener: los cambios en la energía libre (ΔG), entalpias (ΔH), entropía (ΔS), capacidad calorífica (ΔCp) de varias transiciones de los alimentos. La diferencia estructural y energética entre los estados iniciales y finales de los procesos en alimentos pueden ser medidos por calorimetría. También la calorimetría puede ser usada para evaluar los efectos de otras variables físicas y químicas y comparar los termogramas de los materiales antes y después de exponer las variables fuera del calorímetro (Crispín y col., 2015).

3.8.3 Importancia de la evaluación sensorial

La evaluación sensorial es la disciplina científica utilizada para medir, analizar e interpretar las reacciones a aquellas características de un determinado producto que son percibidas por los sentidos de la vista, olfato, gusto, tacto y oído. La caracterización sensorial de alimentos es una de las herramientas más utilizadas en la evaluación de los alimentos y permite tener una descripción completa de sus características. Algunas de sus aplicaciones son control de calidad, desarrollo y estabilidad de productos, procesos y determinación de vida útil. Este tipo de análisis comprende un conjunto de técnicas para la medida precisa de las respuestas humanas a los alimentos y minimiza los potenciales efectos de desviación que la identidad de la marca y otra información pueden ejercer sobre el juicio del consumidor. Es decir, intenta aislar las propiedades sensoriales u organolépticas de los alimentos o productos en sí mismos y aporta información muy útil para su desarrollo o mejora, para la comunidad científica del área de alimentos y para los directivos de empresas (Varela y Ares, 2012).

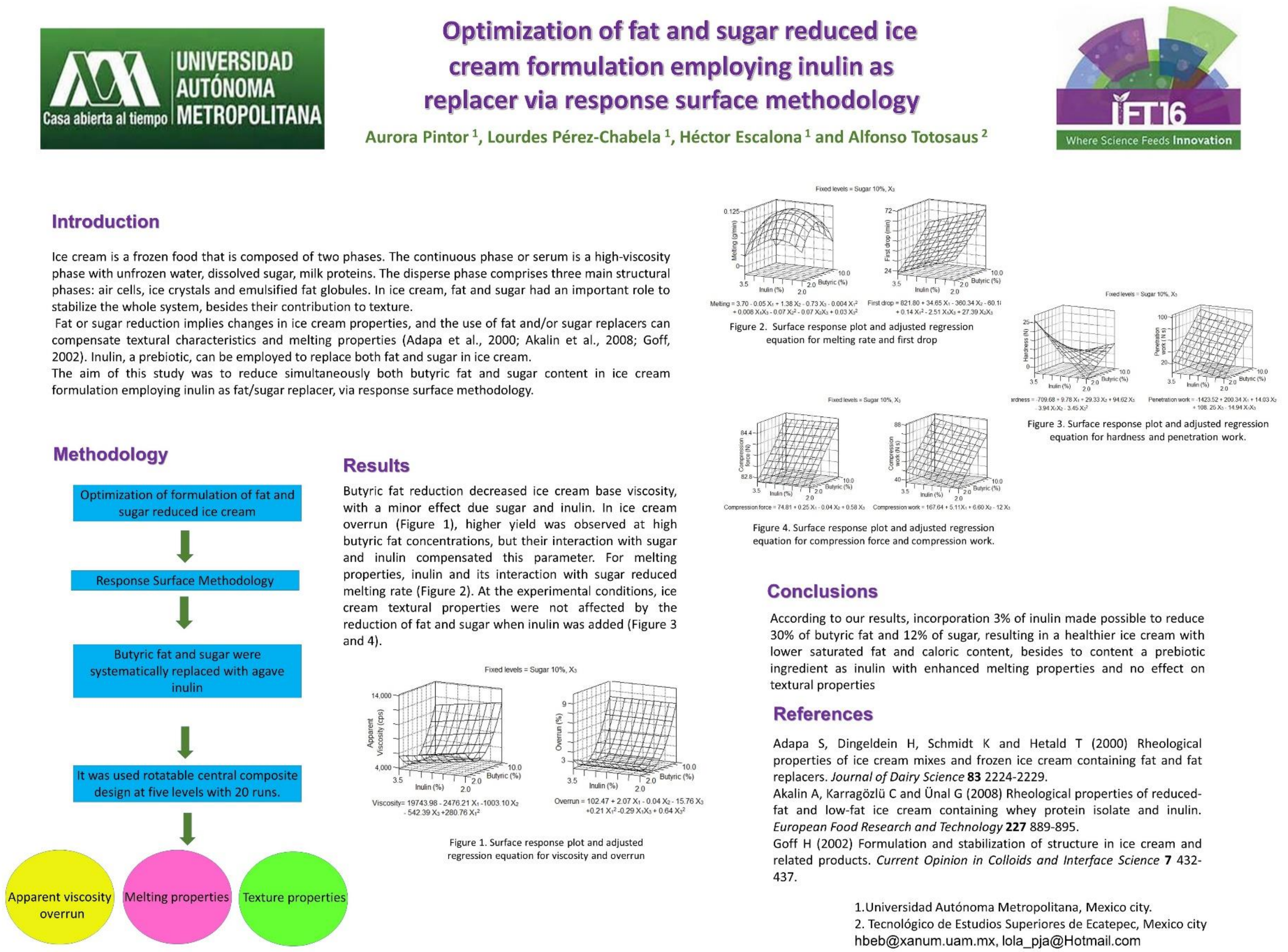
Anteriormente, el análisis sensorial se consideraba como un método marginal para la medición de la calidad de los alimentos. Sin embargo, su desarrollo histórico ha permitido que en la actualidad la aplicación de este análisis en la industria alimentaria sea reconocida como una de las formas más importantes de asegurar la aceptación del producto por parte del consumidor (Varela y Ares, 2012).

4. Artículo 1- Effect of inulin on melting and textural properties of low-fat and sugar reduced ice cream: optimization via a response surface methodology

Introducción.

El siguiente capítulo muestra los resultados de la reducción de grasa y azúcar en un helado, usando inulina de achicoria como remplazo, así como una metodología de superficie de respuesta para optimizar la formulación. Fue implementado un diseño rotable de composición central (15 corridas más 5 repeticiones del punto central) con diferentes niveles de grasa butírica, azúcar e inulina, usando como variables de respuesta viscosidad, *overrun*, propiedades de derretimiento (primera gota y velocidad de derretimiento) y de textura (pruebas de penetración y compresión).

Como resultado se obtuvo una viscosidad aparente mayor cuando se incrementó la concentración de inulina, lo cual afecto positivamente en la cantidad de aire incorporada en las muestras (*overrun*) durante el batido. La mejora de las propiedades de derretimiento reflejó el estado estable de la matriz, cristales de hielo-grasa emulsionada con las burbujas de aire, producto de la interacción de la inulina y otros compuestas con el medio acuoso. Los resultados de textura mostraron que el uso de inulina provocó una mayor retención de agua libre cuando la grasa y azúcar fueron reducidos, lo que dio como resultado cristales de hielo más pequeños y por lo tanto valores de penetración y compresión menores. En las condiciones experimentales propuestas, se pudo emplear inulina (3%) para reducir un 30% de contenido de grasa butírica y un 12% de contenido de azúcar.



Optimization of fat and sugar reduced ice cream formulation employing inulin as replacer via response surface methodology

Aurora Pintor [1], Lourdes Pérez-Chabela [1], Héctor Escalona [1] and Alfonso Totosaus [2]

Introduction

Ice cream is a frozen food that is composed of two phases. The continuous phase or serum is a high-viscosity phase with unfrozen water, dissolved sugar, milk proteins. The disperse phase comprises three main structural phases: air cells, ice crystals and emulsified fat globules. In ice cream, fat and sugar had an important role to stabilize the whole system, besides their contribution to texture.

Fat or sugar reduction implies changes in ice cream properties, and the use of fat and/or sugar replacers can compensate textural characteristics and melting properties (Adapa et al., 2000; Akalin et al., 2008; Goff, 2002). Inulin, a prebiotic, can be employed to replace both fat and sugar in ice cream.

The aim of this study was to reduce simultaneously both butyric fat and sugar content in ice cream formulation employing inulin as fat/sugar replacer, via response surface methodology.

Methodology

Optimization of formulation of fat and sugar reduced ice cream

Response Surface Methodology

Butyric fat and sugar were systematically replaced with agave inulin

It was used rotatable central composite design at five levels with 20 runs.

Apparent viscosity overrun

Melting properties

Texture properties

Results

Butyric fat reduction decreased ice cream base viscosity, with a minor effect due sugar and inulin. In ice cream overrun (Figure 1), higher yield was observed at high butyric fat concentrations, but their interaction with sugar and inulin compensated this parameter. For melting properties, inulin and its interaction with sugar reduced melting rate (Figure 2). At the experimental conditions, ice cream textural properties were not affected by the reduction of fat and sugar when inulin was added (Figure 3 and 4).

$$\text{Viscosity} = 19743.98 - 2476.21\,X_1 - 1003.10\,X_2 - 542.39\,X_3 + 280.76\,X_1^2$$

$$\text{Overrun} = 102.47 + 2.07\,X_1 - 0.04\,X_2 - 15.76\,X_3 + 0.21\,X_1^2 - 0.29\,X_1X_3 + 0.64\,X_3^2$$

Figure 1. Surface response plot and adjusted regression equation for viscosity and overrun

$$\text{Melting} = 3.70 - 0.05\,X_1 + 1.38\,X_2 - 0.73\,X_3 - 0.004\,X_1^2 + 0.008\,X_1X_3 - 0.07\,X_2^2 - 0.07\,X_2X_3 + 0.03\,X_3^2$$

$$\text{First drop} = 821.80 + 34.65\,X_1 - 360.34\,X_2 - 60.1 + 0.14\,X_1^2 - 2.51\,X_1X_3 + 27.39\,X_2X_3$$

Figure 2. Surface response plot and adjusted regression equation for melting rate and first drop

$$\text{ardness} = -709.68 + 9.78\,X_1 + 29.33\,X_2 + 94.62\,X_3 - 3.94\,X_1X_2 - 3.45\,X_3^2$$

$$\text{Penetration work} = -1423.52 + 200.34\,X_1 + 14.03\,X_2 + 108.25\,X_3 - 14.94\,X_1X_3$$

Figure 3. Surface response plot and adjusted regression equation for hardness and penetration work.

$$\text{Compression force} = 74.81 + 0.25\,X_1 - 0.04\,X_2 + 0.58\,X_3 \qquad \text{Compression work} = 167.64 + 5.11\,X_1 + 6.60\,X_2 - 12\,X_3$$

Figure 4. Surface response plot and adjusted regression equation for compression force and compression work.

Conclusions

According to our results, incorporation 3% of inulin made possible to reduce 30% of butyric fat and 12% of sugar, resulting in a healthier ice cream with lower saturated fat and caloric content, besides to content a prebiotic ingredient as inulin with enhanced melting properties and no effect on textural properties

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Figura 12. Congreso internacional, IFT, presentación de poster "Fat and sugar reduction in ice cream employing inulin as fat and sugar replacer". Chicago Illinois, USA.

International Food Research Journal 24(4): 1728-1734 (August 2017)

Effect of inulin on melting and textural properties of low-fat and sugar-reduced ice cream: optimization via a response surface methodology

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Article history

Received: 25 July 2016 Received in revised form: 23 August 2016 Accepted: 24 August 2016

Keywords

Low-fat
Sugar-reduced
Inulin
Ice cream
RSM
Optimization

Abstract

Ice cream is a dairy product with relatively higher fat and sugar content. In this work the simultaneously reduction of both butyric fat and sugar content in ice cream formulation via a response surface methodology was investigated. A rotatable central composite design (15 runs plus 5 central point replications) with different butyric fat, sugar and inulin content was employed to study the effect on overrun, viscosity, melting and textural properties of ice cream. Higher apparent viscosity resulted in a more stable system with higher overrun, where inulin controlled available water. The improvement in melting properties reflected the stable state of the air bubbles-emulsified fat-ice crystals matrix, where the putative effect of inulin to retain water compensating solids and fat reduction, retarded ice crystals melting. In instrumental texture, inulin retained free water when butyric fat and sugar were reduced, resulting in smaller ice crystals reflecting a softer texture. At the experimental conditions proposed, inulin (3%) as functional ingredient (soluble fiber and prebiotic) can be employed to reduce 30% butyric fat content and 12% sugar content, in the formulation of low-fat reduced-sugar ice cream.

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Introduction

Ice cream is a complex food system with a disperse phase consist in three main structural components (air bubbles, ice crystals and emulsified fat globules) immersed in a continuous liquid phase (unfrozen water with dissolved sugar, proteins and hydrocolloids). Fat and sugar are the compounds that provide the caloric content in ice cream. Reduce or replace these important ingredients from ice cream formulations mainly affect the texture and sensorial perception (Clarke, 2004). Fat had many functions on physicochemical and sensorial properties in ice cream, for example: it is responsible of emulsion formation, increase the viscosity of serum phase, creates a film on the surface of the air cells that promote the stability in the ice cream, decrease the melting time, reduce the growth and size of ice crystals, provide texture, palatability, creaminess, releases flavor molecules and enhances mechanical properties (Goff, 1997; Adapa *et al*., 2000; Goff, 2002). Sugars in ice cream had two principal functions: provide sweetness and they control the amount and size of ice crystals, affecting ice cream softness. Sugar has the ability to decrease the freezing point of serum phase and therefore reduce the amount of ice caused for crystallization and recrystallization. The high molecular weight of sugars increase the viscosity of serum phase, promoting an increase of trap air, slow melting and low hardness of ice cream (Hagiwara and Hartel, 1996; Goff and Flores, 1999).

Obesity and overweight in many countries of the world have risen sharply over the past two decades, involving altered eating habits or the increasingly sedentary lifestyles, and the sharpest change in diet structure has involved added sugars and fat. Even low income families have the highest rates of overweight, due to high palatability and low energy cost of added sugars and fats. Strategies for obesity prevention increasingly focus on fiscal and policy measures to limit the consumption of fats and sweets (Drewnowski, 2003). The technological challenge is then to replace both fat and sugar without detrimental effect on food properties. For example, replacing fat and sugar employing inulin affected textural properties and overall acceptability in cakes (Rodriguez-Garcia *et al*., 2014). Inulin has been employed to replace fat in ice cream, decreasing melting rate, besides to increase adhesiveness and hardness (Akbari *et al*., 2016). In same manner, other gums had been employed in low fat ice cream. Javidi *et al*. (2016) reported that basil seed gum and guar

Table 1. Experimental points design (uncoded, coded) and experimental results for the low-fat sugar-reduced ice cream formulation

Run	% Butyric fat (X_1)	% Inulin (X_2)	% Sugar (X_3)	Apparent viscosity (Cps) (Y_1)	Overrun (%)(Y_2)	Melting rate (g/min) (Y_3)	First drop (min) (Y_4)	Hardness (N) (Y_5)	Compression force (N) (Y_6)
1	4.0 (-0.5)	2.5 (0.25)	10.5 (-0.5)	1920	2.10	0.08	35	5.90	14.1
2	4.0 (-0.5)	3.5 (0.75)	13.5 (0.5)	3920	3.10	0.03	46	14.20	37.0
3	8.0 (0.5)	2.5 (0.25)	13.5 (0.5)	7040	5.00	0.10	50	6.00	17.3
4	8.0 (0.5)	3.5 (0.75)	10.5 (-0.5)	8320	5.00	0.12	30	7.00	16.8
5	6.0 (0.0)	2.0 (0.0)	9.0 (0.0)	4320	3.40	0.11	40	9.90	26.3
6	6.0 (0.0)	2.5 (0.0)	12.0 (0.0)	5120	3.20	0.12	45	10.10	11.7
7	4.0 (-0.5)	2.5 (0.25)	13.5 (0.5)	3680	3.20	0.09	38	5.70	9.4
8	4.0 (-0.5)	3.5 (0.75)	10.5 (-0.5)	6880	5.00	0.06	42	7.90	23.7
9	8.0 (0.5)	2.5 (0.25)	10.5 (-0.5)	6400	4.20	0.08	55	8.30	11.1
10	8.0 (0.5)	3.5 (0.75)	13.5 (0.5)	13930	10.00	0.04	65	4.40	7.4
11	6.0 (0.0)	2.0 (0.0)	15.0 (1.0)	7200	3.10	0.07	30	7.80	15.0
12	6.0 (0.0)	4.0 (0.0)	12.0 (-1.0)	5600	4.00	0.08	40	5.60	7.4
13	10.0 (1.0)	2.0 (0.0)	12.0 (0.0)	5130	2.80	0.06	40	9.70	9.6
14	2.0 (-1.0)	2.0 (0.0)	12.0 (0.0)	8640	2.80	0.11	60	4.80	4.4
15	6.0 (0.0)	2.0 (1.0)	12.0 (0.0)	6080	2.40	0.08	40	13.30	11.4
16	6.0 (0.0)	2.0 (0.0)	12.0 (0.0)	4720	4.20	0.08	50	9.30	13.1
17	6.0 (0.0)	2.0 (0.0)	12.0 (0.0)	5440	2.80	0.12	45	9.00	14.4
18	6.0 (0.0)	2.0 (0.0)	12.0 (0.0)	5520	2.60	0.12	45	14.00	14.0
19	6.0 (0.0)	2.0 (0.0)	12.0 (0.0)	5600	3.80	0.13	45	9.00	12.2
20	6.0 (0.0)	2.0 (0.00)	12.0 (0.0)	5320	3.35	0.15	46	10.35	13.4

gum favored the perception of creaminess, depressed coldness and coarseness perception. Basil seed gum reduced meltdown rate and can be employed as fat replacer/stabilizer in low fat ice cream. Also chia seed mucilage was employed as ice cream stabilizer, improving overrun, texture and melting rate (Campos *et al*., 2016). Nonetheless, the prebiotic capacity of inulin (Cruz *et al*., 2009; Criscio *et al*., 2010), besides their techno-functional properties as fat replacer in dairy products (Meyer *et al*., 2011; Tiwari *et al*., 2014), made inulin the better option to formulate low fat and sugar reduced ice cream, with added prebiotic. In low-calorie functional ice cream inulin increased overrun and hardness, besides to improve the viability of B. lactic, where melting rate and sensory scores of low fat and/or low-sugar formulation (Hashemi *et al*., 2014). In same manner, Fragoso *et al*. (2016) reported that thermotolerant lactic acid bacteria improved sensory, melting and textural properties of low-fat ice cream formulated with inulin.

The aim of this study was to reduce simultaneously butyric fat and sugar content, employing inulin to compensate fat and sugar, via response surface methodology, and the effect of formulation on apparent viscosity, overrun, textural and melting properties.

Material and Methods

Ice cream formulation

Fat-reduced ice cream was elaborated according the formulation described by Pintor and Totosaus (2012). Solid ingredients like sugar (15% w/v), non-fat dry milk and whey protein concentrate (8.0 and 4.0% w/v, respectively, DILAC S.A de C.V.), emulsifier (sorbitan and glyceryl monostearates, 0.25% w/v, ARCY S.A. de C.V. Ecatepec, México) were hydrated in water (aprox. 58% v/v) at 60°C to disperse anhydrous butyric fat (10% w/v, ARCY S.A. de C.V., Ecatepec, México) and vegetable fat (4% w/v, La Mixteca, Ecatepec, México). Agave inulin (Vaserco, S.R.L. de C.V., Guadalajara, México) was employed to replace both fat and sugar. The homogenized ice cream base was pasteurized at 70°C for 30 min, cooled down to 4°C in ice bath, and stored at 2-4°C during 24 h. The ice cream base was frozen in a 2 quarters frozen ice cream CIM 50RSA machine (Cuisinart, East Windsor) for 20 min until obtain a uniform frozen paste. Samples were placed in plastic containers (123 mL) and kept frozen at -25°C.

Experimental design, data analysis and optimization

The optimization of the formulation of fat-reduced ice cream to enhance its physicochemical and textural parameters was carried out employing a response surface methodology. Butyric fat and sugar were systematically replaced with agave inulin. A rotatable central composite design was proposed for optimization of ice cream formulation at five levels with 20 runs, including five replicates of central point (Table 1) (Montgomery and Runger, 2010). The experimental results were analyzed in SAS software v. 8.0 ADX interface (SAS Institute, Cary), fitting second order model to establish relationship between independent variables (butyric fat X_1, sugar X_2 and inulin X_3) with response variables Y, as follows:

$$Y = \beta_0 + \Sigma_{i=1}^{k} \beta_i X_i + \Sigma_{i=1}^{k} \beta_{ii} X_i^2 + \Sigma\Sigma_{i<j} \beta_{ij} X_i X_j + \in \qquad (1)$$

Where Y is the response variable that corresponds to physicochemical and textural measures, β_0, β_i, and β_{ij} are the estimate regression coefficients, and $\in$ is the experimental error. Response contour plots were generated in the same software holding one variable constant (butyric fat, central point, 6%).

Optimization of ice cream was performed selecting the desirability of the model for each one of the responses measured in the Desirability function of the Prediction profiler in same SAS ADX interface, where according to SAS support the overall desirability can be defined as the geometric mean of the desirability for each response. Multiple responses in the central composite design were maximized or minimized, according to desirable characteristics in ice cream, i.e., higher viscosity and overrun, slow melting, and a softer texture (Roland *et al*., 1999; Aime *et al*., 2001).

Apparent viscosity and overrun

The apparent viscosity were determined to ice cream base in a Brookfield RVT viscometer (Brookfield Laboratories, Middleboro), adapting the methodology reported by Akesowan (2008). Samples were cooled to 10°C and analyzed with a spindle #07 at 50 rpm after 30 s, reporting ice cream base viscosity in centipoises.

Ice cream yield, overrun, was determined as described by Marshall *et al*. (2003), according to:

$$\% \text{ Overrun} = \frac{(\text{Ice cream weight} - \text{Ice cream base weight})}{\text{Ice cream base weight}} \times 100 \qquad (2)$$

Melting properties

Melting properties were determined according to the report by Soukoulis *et al*. (2008), with some modifications. First drop time and the melting rate were determinate by removing standardized ice cream samples from the containers and putting them on a stainless steel mesh (size 14, 1.41 mm pore size) at room temperature (25±2°C); the time (min) elapsed to obtain the first drop of melting ice cream was registered. The weight of the material that passed through the stainless steel mesh was recorded at 5 min time intervals during 1 h to obtain the melting rate (weight change per minute, according to the slope of the dripped portion as function of the time, in g/min).

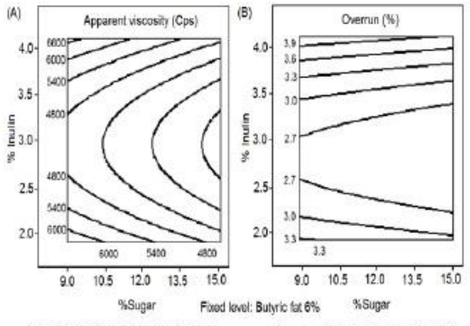

Figure 1. Contour plot and adjusted regression equation for (a) viscosity and (b) overrun (X_1: Butyric fat, X_2: Inulin, X_3: sugar).

Textural properties

The hardness of the ice cream was determined according to the method described by Soukoulis *et al*. (2008). Samples in plastic containers (123 mL) were temper at room temperature for 20 min and penetrated 8 mm from surface with a 10 mm diameter acrylic probe at a constant speed of one mm/s with a Brookfield LFRA 4500 texture analyzer (Brookfield Laboratories, Middleboro), reporting hardness as the peak force during penetration.

For the compression test, the methodology reported by Clark (2004) was adapted. Ice cream base was frozen in PVC cylindrical molds (15.70 cm2 diameter and 2.0 cm height) to form solid ice cream cylinders. Molds were removed and samples were compressed between two 10 cm diameter acrylic plates 40% of the original height in same the Brookfield texture analyzer at a constant rate of 1 mm/s. From force-deformation curves, compression force (maximum load peak) was calculated.

Results and Discussion

Apparent viscosity and overrun

According to the apparent viscosity results, the adjusted second-order model was highly significant (P=0.0001) and had a high correlation coefficient (R^2=0.8435). It was observed that the butyric fat linear term (X_1) and the butyric fat quadratic term

(X_1^2) had a highly significant effect ($P<0.01$) on this parameter. Inulin (X_2) and its quadratic term (X_2^2) presented a significantly ($P<0.05$) effect. No significantly ($P>0.05$) of sugar (X_3) was observed on ice cream base viscosity. In the regression equation, the positive sign of the linear and quadratic terms for butyric fat showed that apparent viscosity values increased mainly due to butyric fat effect, and the quadratic term for inulin (positive sign as well) (Figure 1a). In the contour plot it can be appreciated, at a fixed butyric fat level (6.0%), how at higher inulin concentrations and lower sugar content ice cream base the apparent viscosity increased (Figure 1a).

Fat that is dispersed and distributed through the continuous phase plays an important role in the increase of viscosity in ice-cream. When fat is reduced in the ice cream base, two phenomena are observed: a decrease in base viscosity due to a lower volume of fat aggregates in the continuous phase; and the decrease in formation of a film of fat globules on the surface of the air bubbles, reducing stability during melting (Chung and Grün, 2003). At the experimental conditions employed, when butyric fat was reduced together with sugar, inulin compensate the ice cream viscosity base, due to its capacity to form micro-crystals that interact with each other, creating small aggregates that trap water and increase the viscosity of the base (Akalin and Erişir, 2008; Akalin *et al*., 2008; Karaca *et al*., 2009). In this view, butyric fat reduction was compensated by inulin at reduced sugar contents.

Based on the results for overrun, the adjusted second-order analysis had a highly significant effect ($P=0.0001$) on this parameter, with a high correlation coefficient ($R^2=0.9520$). According to the ANOVA, the butyric fat linear term (X_1), butyric fat quadratic term (X_1^2), butyric fat×inulin interaction (X_1X_2), and the inulin quadratic term (X_2^2) had a highly significant effect ($P<0.01$) on this parameter. Inulin (X_2) presented a significantly ($P<0.05$) effect on overrun. No significantly ($P>0.05$) effect of sugar (X_3) was observed. In the regression equation it was found that butyric fat, with a positive sign, both linear and quadratic terms, increased the overrun values of ice-cream. Inulin quadratic term presented as well a positive influence on ice cream overrun (Figure 1b). The contour plot for overrun (Figure 1b), higher inulin concentrations increased overrun, at a fixed butyric fat level (6.0%), at lower sugar concentration.

High overrun were related to higher viscosities that promote more efficient air incorporation and the formation of smaller air cells (Chang and Hartel, 2002; Akin *et al*., 2007). In low-fat ice-creams where inulin was added, an increase in overrun values reported was related to the high viscosities caused by inulin (Akalin *et al*., 2008). At the experimental conditions, butyric fat reduction was then compensated with inulin, probably enhancing the air bubbles stability during and after freezing process, even at reduced solids (sugar) content.

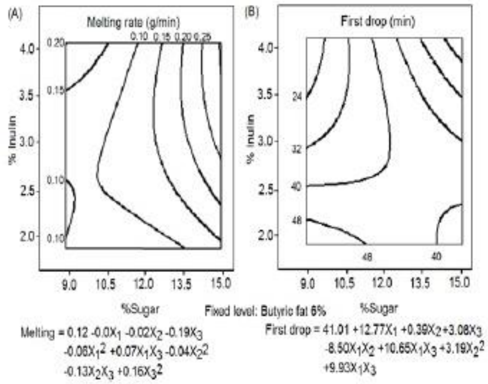

Figure 2. Contour plot and adjusted regression equation for (a) melting rate and (b) first drop (X_1: Butyric fat, X_2: Inulin, X_3: sugar).

Melting properties

The adjusted second-order model had a highly significant ($P=0.0017$) effect and a high correlation ($R^2=0.8426$) for the melting rate. Based on the ANOVA results, inulin (X_2), sugar (X_3), butyric fat quadratic term (X_1^2) and sugar quadratic term (X_3^2) had a highly significantly ($P<0.01$) effect on this parameter. Butyric fat (X_1), inulin quadratic (X_2^2), butyric fat×sugar (X_1X_3) interaction, and inulin×sugar (X_2X_3) interaction presented a significantly ($P<0.05$) effect. In regression equation, most of the parameters presented negative sign meaning that the increase in there ingredients concentration decreased melting rate (Figure 2a). In the contour plot (Figure 2a), at a butyric fat fixed level (6.0%), how higher sugar concentrations increased melting rate, decreasing when sugar content decreased with the incorporation of inulin as solids compensator.

Melting properties are influenced by the three structural components that make up the dispersion phase of ice-cream: ice, air and fat. The amount of ice crystals formed depends on the freezable water and therefore on the amount of solids in the ice-cream. Since both butyric fat and sugar were reduced, according to the experimental design proposed, the inulin added to compensate solutes enhancing melting rate. This was also related to apparent viscosity and overrun values, since air bubbles act as an insulating medium that prevents rapid heat transfer from the medium to the ice crystals (Marshall *et al*., 2003; Sofjan and Hartel, 2004). The more air is incorporated (higher overrun), the slower the melting will be (Chang and Hartel, 2002; Caillet *et al*., 2003). Butyric fat had a marked effect on melting, and its interactions with sugar and inulin were related to the complex interactions during free water freezing. Fat and sugar were reduced and inulin incorporation reduced the melting rate of ice cream.

For the first drop fall during melting test, the adjusted second-order model had a highly significant effect (P=0.0001) and a good correlation (R^2= 0.8986). In ANOVA, butyric fat (X_1), butyric fat×inulin (X_1X_2) interaction and inulin×sugar (X_2X_3) interaction presented a highly significantly ($P<0.01$) effect on the time to first drop fall. Inulin (X_2), sugar (X3), inulin quadratic term (X_2^2), and the three interactions (X_1X_2, X_1X_3, and X_2X_3) present a significantly ($P<0.05$) effect on this parameter. According to the proposed regression equation, butyric fat had the stronger positive effect, where higher butyric fat content extend first drop time. Only butyric fat×inulin interaction had negative sign, i.e., their interaction decreased first drop times (Figure 2b). In contour plot, at a butyric fat fixed level (6.0%), it can be observed that at lower sugar concentrations at low inulin concentration, the first drop time increased (Figure 2b).

Reducing fat increased the melting rate of ice-cream (Roland *et al*., 1999; Akalin *et al*., 2008), but in reduced-fat ice-cream, the inulin acts as a stabilizer, due to its capacity to retain and immobilize large amounts of water, causing less crystallization and longer melting time (Caillet *et al*., 2003; Muse and Hartel, 2004; Meyer *et al*., 2011). Again, the stabilizing property of the inulin compensates the fat and solids reduction. Butyric fat presented as well the most marked effect on time for first drop fall, and the interaction of this ingredient with inulin and sugar on melting represents the interaction between all the soluble compounds during emulsion formation and ice crystals formation and stabilization. Butyric fat and sugar were counterbalance by inulin to extend the time for first drop fall.

Textural properties

The adjusted model for the hardness of the formulated ice-cream had a significantly (P=0.0108) effect and a high correlation (R^2=0.6763). Based on the ANOVA, only the butyric fat (X_1) linear term presented a highly significantly ($P<0.01$) effect on the model. Inulin (X_2), sugar (X_3), both quadratic terms for butyric fat (X_1^2) and sugar (X_3^2), and butyric fat×sugar (X_1X_3) interaction presented a significantly ($P<0.01$) effect. In the regression equation, butyric fat and sugar concentration had an opposite effect on ice cream hardness. Since sugar linear term was positive, and its quadratic interaction negative, a reprimand effect of sugar on ice cream hardness was observed (Figure 3a). In contour plot, this effect can be observed since at lower sugar concentrations ice cream hardness remained practically constant at the studied inulin concentrations range, at a fixed butyric fat level (6.0%) (Figure 3a).

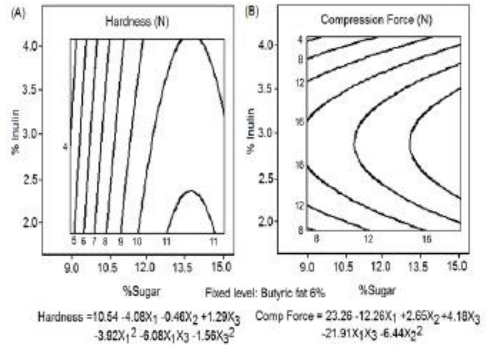

Figure 3. Contour plot and adjusted regression equation for (a) hardness and (b) compression force (X_1: Butyric fat, X_2: Inulin, X_3: sugar).

The model for compression force had a significantly (P=0.0204) effect and a high correlation (R^2=0.6977). The ANOVA showed that linear term for butyric fat (X_1) and butyric fat×sugar (X_1X_3) interaction presented a highly significant ($P<0.01$) effect on compression force, whereas both inulin (X_2) and sugar (X_3) linear terms and inulin quadratic term (X_2^2) had a significant effect ($P>0.05$) on this parameter. Regression equation presented negative sign for butyric fat, butyric fat×sugar interaction and inulin quadratic term (X_2^2) (Figure 3b). This means that butyric fat (and its interaction with sugar) affected the force necessary to compress the samples, but the most marked effect was provoked by inulin. Contour plot shown that an increase in inulin concentration and the decreased in sugar concentration, at a fixed butyric fat level (6.0%), reduced the force necessary to compress the samples (Figure 3b).

The two texture tests serve to determine the uniformity of the structure formed in the ice-cream. Penetration measures the hardness (force required to break the structure of the ice crystals, air bubbles and emulsified fat globules) of the ice-cream and compression test simulate the deformation that occurs during mastication between the palate and the tongue (Clarke, 2004). On reducing the amount

Table 2. Factors setting in ice cream formulation optimization (overall desirability =50.19%)

Factor	Response	Estimate value
Butyric fat= 7.0%	Apparent viscosity	4635.65
Inulin= 3.0%	Overrun	3.45
Sugar= 13.2%	Melting rate	0.0758
	First drop	48.51
	Hardness	8.37
	Compression force	19.71

of fat and solids a harder texture would be expected. When fat content is decreased and compensated with water, then ice crystals were larger, as the ice-phase volume was higher because of the increased water content. In same manner, as the amount of solutes decreases, the ice-phase volume increases, resulting in larger ice crystals, and a harder texture (Hartel, 1996; Clarke, 2004). The use of inulin, a branched polysaccharide of high molecular weight and higher capacity of interaction during ice crystallization, as compared to sugar, decrease ice-phase volume (Goff *et al*., 1993). Incorporation of inulin reduced water molecules mobility from the bulk aqueous phase to the ice crystal surface, resulting in a softer ice cream texture (Soukoulis *et al*., 2009). Texture is the result of components interaction during ice cream freezing, this is, ice crystals formation by the emulsified fat globules and air bubbles matrix. Since inulin compensate on one hand the butyric fat reduction (added water was retained by branched inulin), and on the other hand, sugar reduction (inulin as hygroscopic material that at lower concentration reduce freezable water), the resulting texture was softer (less hard and easy to compress) at lower sugar concentration with relatively low inulin content (around 3%).

Optimization of inulin, sugar and butyric fat levels to enhance textural and melting properties, looking for a softer ice cream texture with longer melting times, is shown in Table 2. Employing inulin (3%) as fat and sugar replacer, butyric fat content can be reduced from 10 to 7% (this is, 30%), and sugar can be reduced as well from 15 to 13.2% (this is, 12%).

Conclusion

Fat and sugar represents two main ingredients in ice cream formulation to define their physical characteristics. Higher apparent viscosity resulted in a more stable system with higher overrun, where inulin controlled available water. The improvement in melting properties reflected the stable state of the air bubbles-emulsified fat-ice crystals matrix, where the putative effect of inulin to retain water compensating solids and fat reduction, retarded ice crystals melting. In instrumental texture, inulin retained free water when butyric fat and sugar were reduced, resulting in smaller ice crystals reflecting a softer texture. At the experimental conditions proposed, inulin as functional ingredient (soluble fiber and prebiotic) can be employed to reduce 30% butyric fat content and 12% sugar content, in the formulation of low-fat reduced-sugar ice cream.

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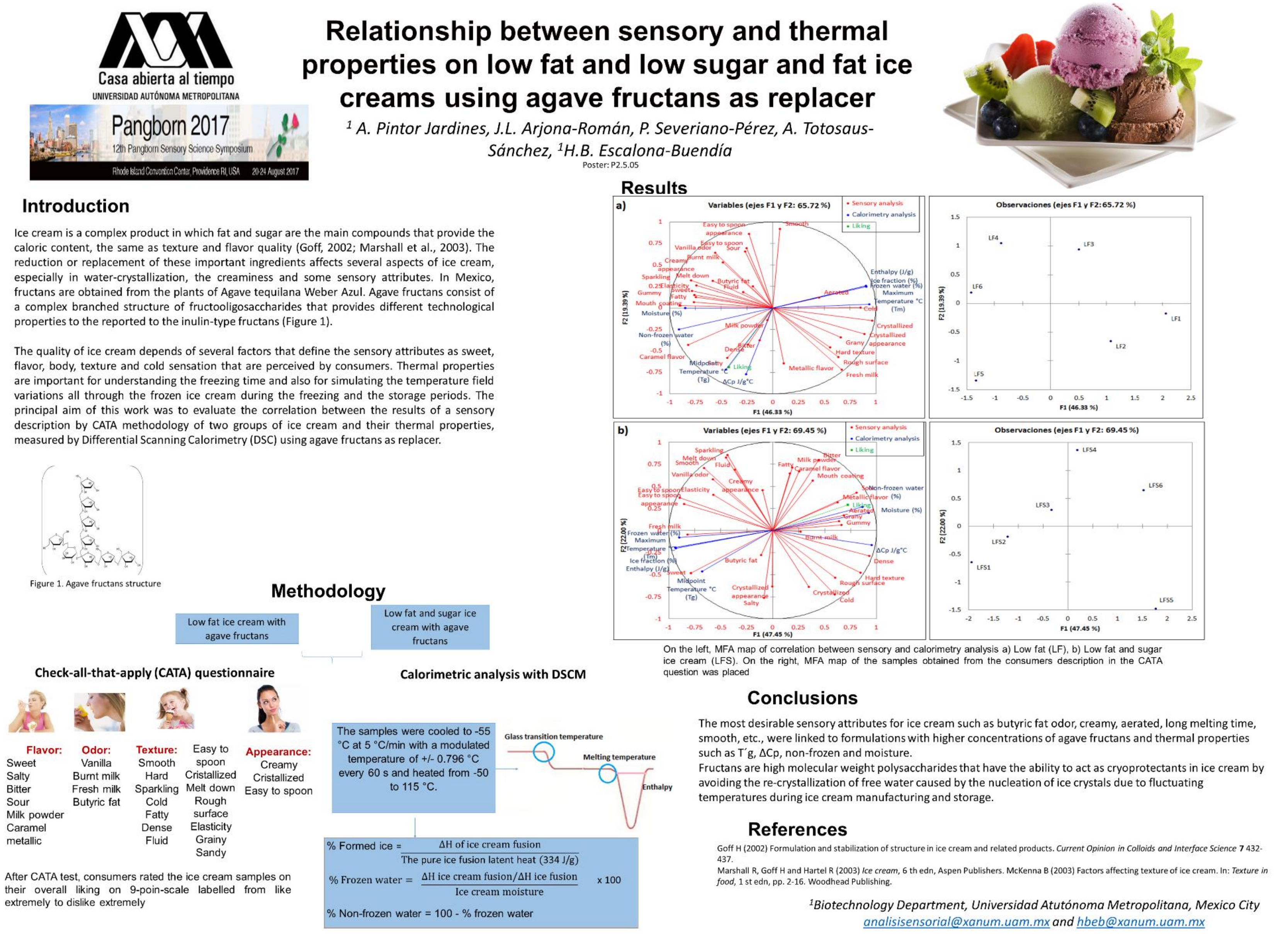
5. Artículo 2- The influence of agave fructans on thermal properties of low-fat, and low-fat and sugar ice cream.

Introducción.

Este capítulo desglosa los resultados del análisis térmico de dos grupos de formulaciones, helados reducidos en grasa y helados reducidos en grasa y azúcar ambos con fructanos de agave como remplazo. Por medio de la técnica de Calorimetría diferencial de barrido se obtuvieron curvas de enfriamiento y calentamiento, para evaluar el uso de 0-3% de fructanos de agave, promoviendo un efecto positivo sobre los dos grupos de formulaciones. Las formulaciones con concentraciones cercanas al 3% obtuvieron una menor concentración de agua congelada y temperaturas de transición vítrea mayores que provocaron un cambio en la capacidad calorífica a medida que aumentaban la humedad y los fructanos de agave.

Debido a la capacidad que tienen los fructanos de agave de reducir la cantidad de agua libre, la formación de cristales de hielo también se vio afectada positivamente y por ende los tiempos de fusión del helado se prolongaron. Las muestras con mayor tiempo de fusión resultaron en una reducción de energía, mostrando valores bajos de entalpía. La cantidad de fructanos de agave utilizados como sustituto de la grasa y el azúcar afectó los espectros de espectroscopia infrarroja, y condujo a un aumento en la magnitud de las bandas, especialmente en el grupo O-H que corresponde a enlaces de hidrógeno polimérico entre fructanos de agave y agua.



Relationship between sensory and thermal properties on low fat and low sugar and fat ice creams using agave fructans as replacer

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Poster: P2.5.05

Introduction

Ice cream is a complex product in which fat and sugar are the main compounds that provide the caloric content, the same as texture and flavor quality (Goff, 2002; Marshall et al., 2003). The reduction or replacement of these important ingredients affects several aspects of ice cream, especially in water-crystallization, the creaminess and some sensory attributes. In Mexico, fructans are obtained from the plants of Agave tequilana Weber Azul. Agave fructans consist of a complex branched structure of fructooligosaccharides that provides different technological properties to the reported to the inulin-type fructans (Figure 1).

The quality of ice cream depends of several factors that define the sensory attributes as sweet, flavor, body, texture and cold sensation that are perceived by consumers. Thermal properties are important for understanding the freezing time and also for simulating the temperature field variations all through the frozen ice cream during the freezing and the storage periods. The principal aim of this work was to evaluate the correlation between the results of a sensory description by CATA methodology of two groups of ice cream and their thermal properties, measured by Differential Scanning Calorimetry (DSC) using agave fructans as replacer.

Figure 1. Agave fructans structure

Methodology

Low fat ice cream with agave fructans

Low fat and sugar ice cream with agave fructans

Check-all-that-apply (CATA) questionnaire

Flavor:
- Sweet
- Salty
- Bitter
- Sour
- Milk powder
- Caramel
- metallic

Odor:
- Vanilla
- Burnt milk
- Fresh milk
- Butyric fat

Texture:
- Smooth
- Hard
- Sparkling
- Cold
- Fatty
- Dense
- Fluid
- Easy to spoon
- Cristallized
- Melt down
- Rough surface
- Elasticity
- Grainy
- Sandy

Appearance:
- Creamy
- Cristallized
- Easy to spoon

After CATA test, consumers rated the ice cream samples on their overall liking on 9-poin-scale labelled from like extremely to dislike extremely

Calorimetric analysis with DSCM

The samples were cooled to -55 °C at 5 °C/min with a modulated temperature of +/- 0.796 °C every 60 s and heated from -50 to 115 °C.

$$\%\ \text{Formed ice} = \frac{\Delta H \text{ of ice cream fusion}}{\text{The pure ice fusion latent heat (334 J/g)}}$$

$$\%\ \text{Frozen water} = \frac{\Delta H \text{ ice cream fusion}/\Delta H \text{ ice fusion}}{\text{Ice cream moisture}} \times 100$$

% Non-frozen water = 100 - % frozen water

Results

On the left, MFA map of correlation between sensory and calorimetry analysis a) Low fat (LF), b) Low fat and sugar ice cream (LFS). On the right, MFA map of the samples obtained from the consumers description in the CATA question was placed

Conclusions

The most desirable sensory attributes for ice cream such as butyric fat odor, creamy, aerated, long melting time, smooth, etc., were linked to formulations with higher concentrations of agave fructans and thermal properties such as T´g, ΔCp, non-frozen and moisture.

Fructans are high molecular weight polysaccharides that have the ability to act as cryoprotectants in ice cream by avoiding the re-crystallization of free water caused by the nucleation of ice crystals due to fluctuating temperatures during ice cream manufacturing and storage.

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Figura 13. Congreso Internacional, Pangborn, presentación de poster ”Relationship between sensory and thermal properties on low fat and low fat and sugar ice creams, using agave fructans asreplacer. Verona, Italia.

LWT - Food Science and Technology 93 (2018) 679–685

Contents lists available at ScienceDirect

LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt

The influence of agave fructans on thermal properties of low-fat, and low-fat and sugar ice cream

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ABSTRACT

The thermal properties of low fat, and low fat and sugar ice cream formulations, in which agave fructans were used as a sugar and fat, were evaluated by Modulated Differential Scanning Calorimetry. Approximately 3.0 g/100 mL of agave fructans promoted a positive effect on thermal properties for both types of formulation, showing a significantly lower ($P < 0.05$) fraction of frozen water. The highest concentration of agave fructans resulted in the highest percentage of non-frozen water ($P < 0.05$). The addition of agave fructans led to a significantly higher ($P < 0.05$) glass transition temperature. Glass transition incited a change in heat capacity as moisture and agave fructans increased. Agave fructans had the ability to reduce the number and formation of ice crystals, and hence the melting temperature of ice cream. The samples with longer melting time resulted in a reduction of energy, showing low enthalpy values. The quantity of agave fructans used as fat and sugar replacer affected the infrared spectroscopy spectra, and led to an increase in the magnitude of the bands, especially in the O-H group that corresponds to polymeric hydrogen bonding between agave fructans and water.

1. Introduction

The amount and functionality of ingredients employed in ice cream formulations influence texture, colloidal aspects such as microstructure, viscosity of serum phase, emulsion characteristics and thermal properties that determine the overall quality of the ice cream (Goff, 1997; Marshall, Goff, & Hartel, 2003; Muse & Hartel, 2004; Regand & Goff, 2003). Fat and sugar are the main compounds that provide the caloric content in ice cream. The reduction or replacement of these important ingredients in ice cream formulations affects several quality parameters such as thermal properties (Clarke, 2004).

In several studies, chicory inulin has been used as a fat and sugar replacer, or as a food supplement in combination with water to produce a texture and mouth feel similar to fat in ice cream (Akalin, Karragözlü, & Ünal, 2008; Karaca, Güven, Yaser, Kaya, & Kahyaoglu, 2009; Meyer, Bayarri, Tarrega, & Costell, 2011). In comparison with chicory inulin (fructans with linear structure) agave fructans have shown a higher water absorption. This compound consists of a complex branched structure of fructooligosaccharides linked by β (2-1) bonds but also β (2-6) with glucose molecules (Espinosa & Urias, 2012).

It is expected that the branched structure of agave fructans could provide some technological properties different from the ones reported with the inulin-type fructans. For example, agave fructans have been used in reduced milk-fat yogurts, modifying their microstructural, sensory and rheological properties positively (Crispín, Lobato, Espinosa, Alvarez, & Vernon, 2015).

Previously, we have studied the effect of chicory inulin in low butyric and vegetable fats ice cream (Pintor, Severiano, & Totosaus, 2013), as well as the effect of agave fructans on low fat and sugar ice cream (Pintor, Escalona, & Totosaus, 2017). For both works, parameters such as: overrun, apparent viscosity, texture and melting properties, sensory analysis were used to optimize the formulations. To complement these studies, looking for a better understanding of the interactions of agave fructans with frozen/non-frozen water in the ice cream, we performed the thermal analysis by Modulated differential scanning calorimetry (MDSC) and Fourier transform infrared spectroscopy (FTIR).

Many studies have focused on the physical, chemical and

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https://doi.org/10.1016/j.lwt.2018.03.060
Received 29 August 2017; Received in revised form 2 February 2018; Accepted 23 March 2018
Available online 24 March 2018
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microbiological stability of food that depends on the water content and its interaction with other ingredients. Stickiness is undesirable during ice cream manufacture; it is a phenomenon that may occur when amorphous food is heated or exposed to high humidity. This change may be controlled by the glass transition temperature (Tg), which determines the critical moisture content when stickiness begins to occur (Espinosa & Urias, 2012). MDSC has been used in polymer science to determine different thermal properties in multi-component systems such as ice cream, in which glass and fusion transitions occur due to temperature fluctuations, affecting amorphous and crystalline compounds (Cogné, Caillet, Andrieu, Laurent & Rivoire, 2003; Roos, 2009; Leonard, 2005; Verdonck, Schaap, & Thomas, 1999). On the other hand, Fourier transform infrared spectroscopy (FTIR) has been used in polymers characterization to provide information about structure and composition (Chen & Irudayaraj, 1998; Silverstein & Webster, 1998) and it is able to provide information about the type of bonding that takes place when polymers interact with other components in a food matrix. (Pourfarzad, Najafi, Khodaparast, & Khayyat, 2015). Therefore, the aim of the present work was to evaluate the effect of reducing simultaneously fat and sugar, employing agave fructans as replacer in low fat (LF) and low fat and sugar (LFS) ice cream on thermal properties such as frozen and un-frozen water, glass transition and melting properties, and to explore possible molecular interactions of agave fructans with the ice cream matrix by FTIR.

2. Methodology

2.1. Ice cream elaboration

For ice cream samples, we departed from two formulations optimized via response methodology in previous stages, according overrun, viscosity, melting, textural properties and sensory results: one low fat (LF) and the other low fat and sugar (LFS) (Pintor et al., 2013; Pintor et al., 2017). Then, for this work we developed 12 different ice creams: six equidistant points for the optimum LF and another six equidistant points for the optimum LFS (Table 1).

For ice cream manufacture, the dry ingredients, sugar (15 g/100 mL), non-fat dry milk and whey protein concentrate (8.0 and 4.0 g/100 mL respectively, DILAC S.A de C.V.) and emulsifiers (sorbitan and glycelyl monostearates, 0.25 g/100 mL, ARCY S.A de C.V. Ecatepec, México) were hydrated in water at 60 °C to disperse anhydrous milk fat (referred to as butyric fat) and vegetable fat (10.0 and 4 g/100 mL respectively, La Mixteca, Ecatepec, México). Agave fructans (Vaserco, S.R.L de C.V., Guadalajara, Mexico) were employed to replace both fat and sugar. When they were characterized by Size-Exclusion Chromatograph, polymerization degree (DP) was 17.1 ± 0.2, having a proportion of fructans (DP > 10) at 72.5 g/100 g ± 0.2 and FOS (DP 3-10) of 27.4 g/100 g ± 0.2, with residual sucrose (0.48 g/100 g), glucose (1.88 g/100 g) and fructose (4.94 g/100 g) contents. The homogenized mix was pasteurized at 70 °C for 30 min and stored at 4 °C during 24 h. The ice cream mix (non-frozen mixture) was frozen in a 2-quarters frozen-ice cream CIM 50RSA machine (Cuisinart, East Windsor) for 20 min.

2.2. Modulated differential scanner calorimetry

Heating and cooling thermograms were obtained with a TA Instrument DSC (Series 2920, New Castle DE, USA) and cooled with a refrigerated cooling system (RCS). The curves were analyzed by TA Instrument Universal Analysis 2000 V 4.5A software. The DSC instrument was calibrated with pure indium for cell constant and sapphire for Cp constant. Aliquots of an average of 3.2 mg for LF and 3.4 mg for LFS of ice cream mix (non-frozen mixture) were used. The method was modified from Blond, (1994) as follows: cooling to −55 °C at 5 K/min with a modulated temperature of ± 0.796 °C every 60 s and heating from −50 to 115 °C.

The ice fraction (expressed as percentage relative to 100 g of sample) was determined by integrating the melting curves and dividing the melting enthalpy with the pure ice fusion latent heat ($\Delta H = 334$ J/g) (Soukoulis, Lebesi, & Tzia, 2009).

The percentage of frozen water was calculated according to Aktas, Tülek & Gökalp, (1997); Alvarez, Wolters, Vodovotz & Ji, (2005) as follows:

$$\%\,Fw = \frac{Melting\ enthalpy/Melting\ enthalpy\ pure\ ice}{Total\ moisture\ of\ each\ sample}$$

The percentage of unfrozen water, obtained by subtracting % FW from 100, is an estimation of the bound water present in the ice cream formulations. Those parameters reflect the impact of varying the composition on water crystallization. Glass transition temperatures (Tg) were obtained from DSC heating curves. These temperatures were calculated from baselines before and after the transition. Onset, maximum temperature (Tm) and offset melting temperatures were calculated. The melting enthalpy ΔH was obtained from the DSC melting curves to find the amount of heat emitted (Goff, Caldwell, & Stanley, 1993). For all the analyses three repetitions were obtained.

2.3. Infrared spectrometry

An Infrared Spectrometer Buck 500 (series 125, Walthan, MA, USA) was employed to determine changes in LF and LFS ice cream. The samples of ice cream were previously lyophilized to form thin tablets, using 1 mg of spray-dried sample with 100–300 mg of KBr (which is an ionic compound that transmits along most of the infrared region at a frequency of about 400/cm). The mix was compressed to form a transparent disc. The study was tested in the media spectral region of 4000–400/cm. Each infrared spectrum was recorded for qualitative analysis and its peaks were analyzed to identify different functional groups, which absorbed different amounts of radiation (Nery, Güemes, Meza & Totosaus, 2015).

2.4. Data analysis

A one-way analysis of variance (ANOVA) was used to find the differences between more than two groups of ice cream formulations. When a significant effect ($P < 0.05$) was found using analysis of variance, it was followed by a Duncan multiple comparison test to identify

Table 1
Ice cream Formulations.

Low fat ice cream (LF)				Low fat and sugar ice cream (LFS)			
Treatment	Butyric fat (g/100 mL)	Vegetable fat (g/100 mL)	Inulin (g/100 mL)	Treatment	Butyric fat (g/100 mL)	Sugar (g/100 mL)	Inulin (g/100 mL)
LF1	10	4.0	0	LFS1	10	15	0
LF2	9.4	3.9	0.6	LFS2	9.4	14.77	0.6
LF3	8.8	3.8	1.2	LFS3	8.8	14.55	1.2
LF4	8.2	3.7	1.8	LFS4	8.2	14.32	1.8
LF5	7.6	3.6	2.4	LFS5	7.6	14.10	2.4
LF6	7.0	3.5	3.0	LFS6	7.0	13.2	3.0

Table 2

Frozen and no-frozen water percent for LF.

Treatment	Moisture (g/100 g)	Ice fraction (%)	Frozen water (%)	Non-frozen water (%)
LF1	$57.6^{a} \pm 0.2$	$41.28^{c} \pm 0.05$	$71.67^{c} \pm 0.01$	$28.32^{a} \pm 0.05$
LF2	$57.7^{a} \pm 0.3$	$34.85^{b} \pm 0.2$	$60.34^{b} \pm 0.2$	$39.65^{b} \pm 0.07$
LF3	$57.85^{b} \pm 0.2$	$38.89^{b} \pm 0.12$	$67.22^{c} \pm 1.8$	$32.77^{a} \pm 0.3$
LF4	$57.95^{b} \pm 0.5$	$33.07^{a} \pm 0.06$	$57.07^{b} \pm 0.9$	$42.92^{b} \pm 0.21$
LF5	$58.15^{c} \pm 1.2$	$31.07^{a} \pm 0.1$	$53.44^{a} \pm 0.06$	$46.55^{c} \pm 0.04$
LF6	$58.65^{c} \pm 0.9$	$30.79^{a} \pm 0.8$	$52.50^{a} \pm 0.07$	$47.49^{c} \pm 2.8$

a, b, c Means with the same letter in the same column are not significantly different ($P < 0.05$).

specific differences between pairs of treatments. All the statistical analyses were performed with XLStat software (Addinsoft, 2009).

3. Results and discussion

3.1. Frozen and non-frozen water

As expected, all formulations with different solid content had a significant ($P < 0.05$) effect on ice cream moisture for both low fat (LF), and low fat and sugar (LFS) ice cream formulations. The moisture was calculated based on the amount of water used to reach the final volume for each formulation (1 L). For LF ice cream, samples 5 and 6, with the higher water content, showed a concomitant significantly ($P < 0.05$) lower frozen water percentage (53.44 and 52.50% respectively). The unfrozen water could be entrapped by agave fructans, reducing free water and hence, forming ice. Formulation 5 and 6, with the higher agave fructans content, also presented a significantly ($p < 0.05$) higher non-frozen water percentage (46.55 and 47.49% respectively), compared with formulations containing lower agave fructans content (Table 2).

For LFS ice cream, a similar effect to that of the LF formulations was shown. Only formulation 6, with a higher moisture content, had the lowest frozen water percentage (45.77%), showing a significant ($P < 0.05$) effect. For the non-frozen water percentage, once again, only formulation 6 (54.22%) was significantly ($p < 0.05$) higher than the other samples (Table 3). The water in ice cream can be in either a free or a bound state, depending on the hydrophilic sites of different compounds, in this case polysaccharides. It is known that only free water can undergo possible state transitions such as ice crystallization (Cogné, Caillet, Andrieu, Laurent & Rivoire, 2003). For the ice fraction, the cryoconcentration of the remaining solution was increased continuously for both formulations, causing a decrease in the freezing point; this effect was observable in total heat flow for both formulations (Fig. 1). As the temperature decreased, the freeze concentration phenomenon continued to drop. At −10 °C about 65% of the total water was frozen and this percentage reached 100% at approx. −37 °C.

The fat reduction, compensated for with added water, resulted in higher ice formation in formulated ice cream, but with a compensatory effect of agave fructans, which chemically bonded free water, avoiding excessive ice crystal formation. In Tables 2 and 3, the amount of ice for both formulations (LF and LFS) shows that as the concentration of agave fructans increases, the ice formation decreases. With chicory inulin, ice crystallization was strongly dependent on the percentage of bound water, caused by both the influence of soluble solids in the aqueous phase and by the water-binding effect of fructans, which formed a gel-like network (El- Negar, Clowes, Tudorica, Kuri & Brennan, 2002; Soukoulis et al., 2009). Chicory inulin led to an increase in bound water due to the high OH group content bound to water (Aktas et al.,1997), resulting in a smooth and creamy texture. Low-fat ice cream commonly contains more water and therefore larger ice crystals (Hartel, 1996). Therefore, the addition of chicory inulin has been used in ice cream to reduce fat and sugar due to its capacity to form aggregates that trap free water, decreasing the ice crystal content formed during freezing, which is reflected in textural and sensorial properties (Akalin & Eri ir, 2008; Akin & Kirmaci, 2007; Ismail, Saleh-Al, & Metwalli, 2013; Meyer et al., 2011; Pintor et al., 2013). In another study, the addition of 3 g/100 ml of inulin decreased the percentage of frozen water in both the sucrose system and the ice cream model (Soukoulis et al., 2009). Therefore, it is possible to propose that agave fructans had a similar behavior to chicory fructans during the crystallization of water.

3.2. Glass transition for LF and LFS

Ice cream is a multi-component system that is composed of a mix of sucrose-polysaccharides and proteins, in which glass and fusion transitions occur due to temperature fluctuations that affect amorphous and crystalline compounds (respectively). The glass transition is the passage of a state known as the gummy state, where there is greater mobility of the molecules and therefore less stability, to another state called metastable (several states of equilibrium) characterized by high aggregation and molecular order. The glass transition temperature (Tg) in ice cream is an indirect way of finding its thermodynamic stability during storage at low temperature (Hagiwara & Hartel, 1996). At this point, the structure of the ice cream remains the same as that of the supercooled liquid, but the change in position and orientation of molecules has mostly stopped (Blond, 1994). Glass transition depends on many factors: the amount and/or type of plasticizer, aqueous phase concentration, the molecular weight of solutes, and cooling rate (Hartel, 1996). This transition only occurs in the amorphous fraction of polymers, whose structure is not arranged in an orderly manner. The results of the addition of agave fructans on glass transition temperatures (Tg) in LF ice cream are shown in Table 4. The addition of approx. 3.0 g/100 mL of agave fructans led to a significant increase in glass

Table 3

Frozen and non-frozen water percent for LFS.

Treatment	Moisture (g/100 g)	Ice fraction (%)	Frozen water (%)	Non-frozen water (%)
LFS1	$62.25^{a} \pm 0.3$	$36.33^{c} \pm 1.2$	$58.36^{c} \pm 0.7$	$41.63^{a} \pm 0.02$
LFS2	$62.48^{a} \pm 0.6$	$34.94^{b} \pm 0.4$	$55.92^{c} \pm 0.01$	$44.97^{a} \pm 0.1$
LFS3	$62.70^{a} \pm 0.3$	$33.50^{b} \pm 0.05$	$53.44^{b} \pm 0.04$	$46.55^{b} \pm 0.2$
LFS4	$62.93^{a} \pm 0.7$	$33.20^{b} \pm 2.2$	$52.76^{b} \pm 0.08$	$47.23^{b} \pm 0.05$
LFS5	$63.15^{b} \pm 0.2$	$31.58^{a} \pm 0.6$	$50.01^{b} \pm 0.2$	$49.98^{b} \pm 0.06$
LFS6	$64.05^{b} \pm 0.2$	$29.31^{a} \pm 0.9$	$45.77^{a} \pm 0.02$	$54.22^{c} \pm 0.03$

a, b, c Means with the same letter in the same column are not significantly different ($P < 0.05$).

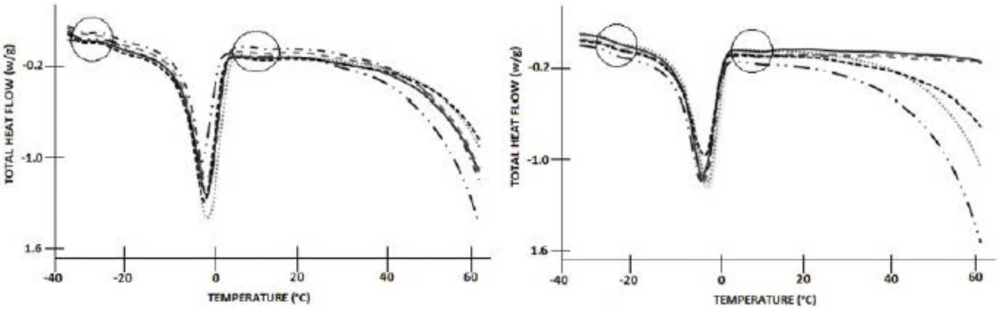

Fig. 1. Total heat flow in heating for LF (left side) and LFS (right side) ice cream samples. Transition zones are indicated in the circles. For both formulations the different type of lines corresponds to each formulation: .... (1), - - - (2), -.-.- (3), – (4), -..-..- (5), —— (6). To each formulation check Table 1.

temperatures, especially in samples 5 and 6, which presented significantly ($P < 0.05$) higher glass transition temperatures (−31.05 and −33.08 °C, respectively) indicating that this formulation started to change from a viscoelastic liquid (rubbery) to an amorphous solid (glass) with increased viscosity. Table 4 shows glass transition temperatures for LFS ice cream. A significant effect ($P < 0.05$) was found for samples 4, 5 and 6, which had higher moisture and agave fructans content. It can be seen that heat capacity increases as glass transition occurs due to the polymers having just gone through the transition. We could infer that agave fructans had a similar behavior to chicory inulin due to the fact that these transitions are very close to chicory inulin transitions of different degrees of polymerization. The glass transition temperature ranged from −43 to 57 °C corresponding to a moisture range from 6 g/100 g–34 g/100 g (dry basis), which is very similar to our formulations (Povolony, Smith, & Labuza, 2000). Glass transition implies a change in heat capacity (Cp) as moisture and agave fructans increase. The samples 5 and 6 for both treatments showed a higher significant effect on ΔCp, indicating that these samples needed more energy to increase the temperature. The increase in temperature is associated with the restriction of the mobility of water molecules and the thermodynamic stability caused by agave fructans. The Tg of ice cream could range from −23 to −43 °C depending on the formulation. Below this temperature range, the ice cream is stable to recrystallization and deteriorative reactions because of the high viscosity (Levine & Slade, 1990). Above Tg, molecular mobility and diffusion increase and viscosity (or elasticity) decreases. In another study, the use of 2.0 or 4.0 g/100 g of chicory inulin provoked an increase in Tg (from −50.5 to −46.6 °C) due to the great influence on the rubbery to glassy transition phenomenon and the barrier against water diffusion and recrystallization (Soukoulis et al., 2009). On the other hand, studies have shown that the glass transition of chicory inulin depends on molecular weight as well as water content. This means that the Tg of chicory inulin decreases with increasing moisture content. Apparently, water functions as a plasticizer since it reduces the inter- and intra-macromolecular forces (Tolstoguzov, 2000; Zimeri & Kokini, 2002).

3.3. *Melting temperature and melting enthalpy for LF and LFS*

The melting resistance in ice cream reflects its ability to resist melting when exposed to higher temperatures and its influence on the function of the compounds (Clarke, 2004). During the DSC heating process, as the temperature continued to increase, an endothermic peak appeared (Tm). Fusion, like crystallization, is a first order thermodynamic transition because it is the first Gibbs derivate to show a sharp increase during the process. For both treatments (LF and LFS), there was a maximal temperature displacement of fusion added to reduction enthalpies from samples 1 to 6. The melting temperatures of both ice cream formulations were significantly ($P < 0.05$) lower for formulations 4 to 6, suggesting that ice cream formulations with higher agave fructans content finish melting after the rest of the samples, and melt more slowly (Table 5). It is likely that agave fructans bind free water and decrease ice crystal formation. It is known that chicory inulin improves ice cream melting properties by forming a three-dimensional network that traps water, provoking a slow melt (Akalin & Erişir, 2008; Karaca et al., 2009a). On the other hand, fat globules are solid at low temperatures, forming a structural network that stabilizes the whole ice cream system, delaying the ice cream melting at room temperature. Nevertheless, when fat content is reduced, and water content –ice crystals– increased, the melted fat did not melt down at ice melting temperatures. The melting enthalpy (ΔH) that was calculated by integrating the area of the melting endotherm peak is the amount of energy removed in a system, and it is manifested as a change in the overall internal energy of food (Roos, 2009). In LF formulations, samples 5 and 6 with lower frozen water content (53.44 and 52.50%, respectively) had the lowest enthalpy (103.80 and 102.85 J/g), showing a significant ($P < 0.05$) effect (Table 5). For LFS ice cream the samples with lower free water content showed a significant effect ($P < 0.05$) in melting enthalpy, causing a reduction in energy (Table 5). A thermal study of chicory inulin demonstrated that the melting enthalpy increases as moisture content rises (Soukoulis et al., 2009; Zimeri & Kokini, 2002). The same effect described above was shown for our samples with agave fructans.

Table 4
Glass transition temperature and ΔCp for LF and LFS samples.

Sample	Midpoint Temperature °C (Tg)	ΔCp (J/g K)	Sample	Midpoint Temperature °C (Tg)	ΔCp (J/g K)
LF1	$-33.98^{ab} \pm 1.98$	$0.83^{c} \pm 0.5$	LFS1	$-33.95^{a} \pm 0.54$	$0.41^{a} \pm 0.5$
LF2	$-34.81^{ab} \pm 1.06$	$0.67^{b} \pm 0.3$	LFS2	$-34.27^{a} \pm 1.88$	$0.32^{a} \pm 0.3$
LF3	$-34.50^{ab} \pm 0.21$	$0.59^{a} \pm 0.1$	LFS3	$-34.50^{b} \pm 0.21$	$0.59^{b} \pm 0.1$
LF4	$-35.45^{b} \pm 1.50$	$0.61^{a} \pm 0.7$	LFS4	$-35.49^{c} \pm 0.51$	$0.61^{b} \pm 0.7$
LF5	$-31.05^{a} \pm 1.51$	$0.96^{c} \pm 0.3$	LFS5	$-34.95^{c} \pm 0.57$	$0.96^{c} \pm 0.3$
LF6	$-33.08^{a} \pm 3.15$	$0.83^{c} \pm 0.6$	LFS6	$-34.75^{c} \pm 0.70$	$0.83^{c} \pm 0.6$

a, b,c Means with the same letter in the same column are not significantly different ($P < 0.05$).

Table 5
Melt temperature and fusion enthalpy for LF and LFS samples.

Sample	Maximum Temperature °C (Tm)	Enthalpy (J/g)	Sample	Maximum Temperature °C (Tm)	Enthalpy (J/g)
LF1	$-5.20^{a} \pm 0.0$	$137.90^{c} \pm 15.38$	LFS1	$-4.61^{a} \pm 0.0$	$121.35^{c} \pm 10.54$
LF2	$-5.33^{a} \pm 0.04$	$116.40^{b} \pm 14.8$	LFS2	$-5.37^{b} \pm 1.01$	$116.70^{c} \pm 9.33$
LF3	$-5.35^{a} \pm 0.0$	$129.9^{c} \pm 0.14$	LFS3	$-5.49^{b} \pm 0.73$	$111.92^{b} \pm 12.8$
LF4	$-6.29^{b} \pm 1.26$	$110.48^{b} \pm 15.21$	LFS4	$-6.30^{c} \pm 0.01$	$110.91^{b} \pm 15.21$
LF5	$-6.29^{b} \pm 1.33$	$103.80^{a} \pm 2.69$	LFS5	$-6.67^{c} \pm 0.04$	$105.50^{b} \pm 14.3$
LF6	$-6.09^{b} \pm 0.03$	$102.85^{a} \pm 13.4$	LFS6	$-6.01^{c} \pm 0.71$	$97.92^{a} \pm 12.80$

a, b,c Means with the same letter in the same column are not significantly different ($P < 0.05$).

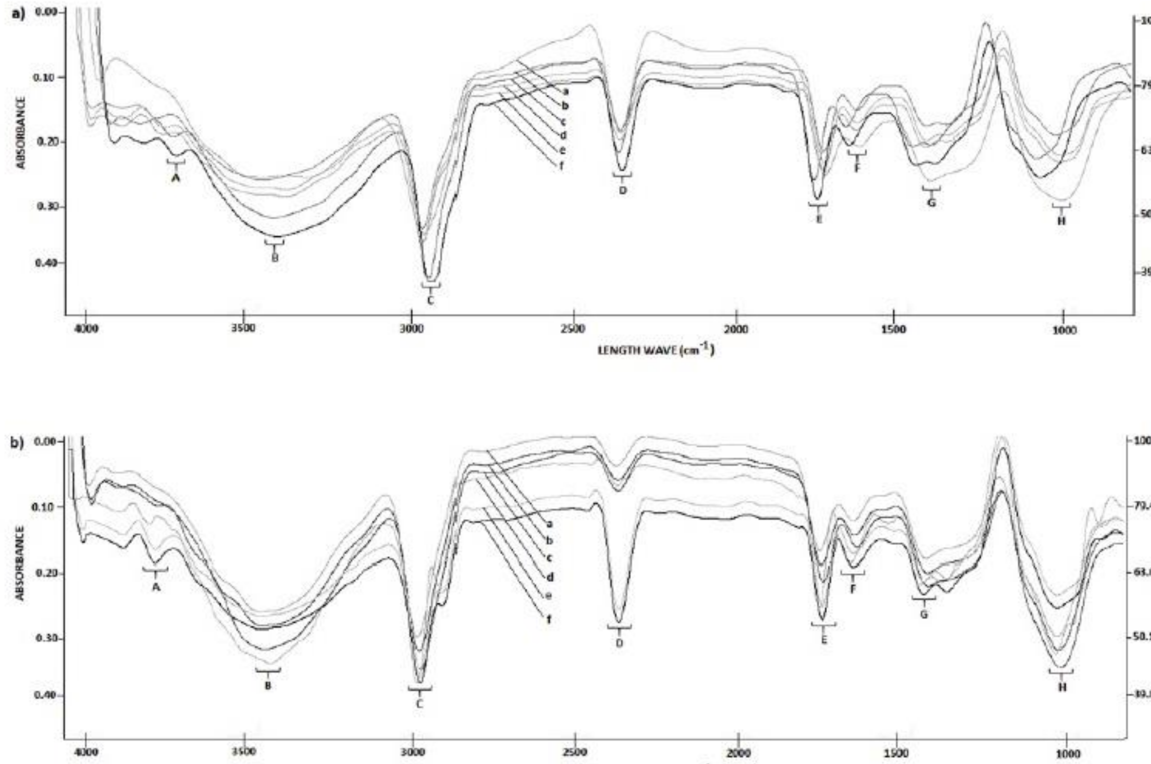

Fig. 2. FTIR spectra for a) low butyric and vegetable fat and b) low fat and sugar with inulin ice cream formulation. The letters correspond different percentage of agave fructans a) 0, b) 0.6, c) 1.2, d) 1.8, e) 2.4, f) 3.0 gr/100 ml. For both treatments: (A) O-H group, non-hydrogen-bonded or "free" (3650-3580/cm), (B) OH group, intermolecular hydrogen bonding (3353/cm), (C) C-H group, fatty acids (2950-3200/cm), (D) R-NH_3^+ group, amine I (2000-2500/cm), (E) C O group, fatty acids (1740/cm), (F) O-H bending group, water (1653/cm), (G) C-N group, amine I & II (1450-1253/cm) and (H) C-O-C group, glucose and glycosidic linkage of inulin (1200-800/cm).

3.4. Fourier transform infrared spectroscopy

There are four relevant zones to study in medium IR: 1. - Stretching vibration region X-H (4000-2500/cm). This absorption corresponds to the extent of hydrogen bonding (alcohols, amines and C-H bonds). The methyl and methylene group with C-H stretching vibrations is the most characteristic and appears between 3000 and 2850/cm. O-H stretching is one of the most dominant and characteristic functional groups and this appears between 3700 and 3600/cm. If there is a hydrogen bond, there is a widening of the bands and a slight decrease in the absorption frequency. N-H stretching is observed between 3400 and 3300/cm. 2. - Triple link region (2500-2000/cm). The functional groups that are absorbed in this zone are (-C N, C O, -C C-, -N+ C-), where C C-stretching is presented as a very weak band. 3. – Double bond region (2000-1550/cm). The principal bands are due to the carbonyl group C O (1830-1650/cm) and C C. Other bands in this area are C N and bending of amines and alcohols. 4. – Fingerprint region (1500-600/cm). Many simple linkages absorb in this region and there is strong interaction between neighboring linkages. It is a little difficult to interpret the spectrum in this region, but allows the usefulness of identification as a "fingerprint". In the medium infrared spectra (MIR) of ice cream, each functional group absorbed radiation and generated an IR band in a particular frequency range. For both formulations LF and LFS (Fig. 2 a, b), the bands correspond approximately to the next functional groups: 3873-3000/cm for O-H stretching vibrations. Non-hydrogen-bonded or "free" water was absorbed strongly in the 3650-3584/cm region. This isolated link has the length and strength of type O-H intermolecular hydrogen bonding as the concentration of the solution increases, and additional bands appear at lower frequencies 3550-3200/cm. These bands correspond to polymeric hydrogen bonding. Different types of O-H bonds with different lengths and bond forces cause wider bands, while weaker long linkages carry lower frequencies (Hesse, Meier, & Zeeh, 2005; Silverstein & Webster, 1998). In the case of H_2O, there are two O-H bonds that would give rise to two stretching modes, a symmetrical and an asymmetrical mode of vibration (3652 and 3756/cm respectively). The other relatively strong absorption band at around 1653/cm reflects the absorption of the O-H bending signal of absorbed water (Pal, Mal, & Singh, 2005). The bands in 2950–3200/cm correspond to C-H group and 1740/cm corresponds to C O, from fatty acids. Other bands near 2000-2500/cm correspond to R-NH3 of amine I group, 1235-1450/cm for C-N of amide I & II group. The bands that are sensitive to the conformation of carbohydrates as glucose appear around 950-1200/cm (Goodfellow & Wilson, 1990) and finally, the absorption of the C-H aliphatic bending from agave fructans is reflected around 600-800/cm. Since there are no results for IR agave fructans, we take chicory inulin as a reference. If we analyze agave fructans versus chicory inulin, the spectra of chicory inulin revealed complex vibrational modes at low wavelength numbers due to the skeletal mode vibrations of the fructose and glucose. The intensity of these wavelength numbers is related to increasing levels of chicory inulin (Kizil, Irudayaraj, & Seetharaman, 2002). Near 1160/cm is related to the C-O-H and glycosidic linkage of chicory inulin (Boussarsar, Rogé, & Mathlouthi, 2007). The samples with a high chicory inulin concentration caused the increase in wave number bands due to the water being trapped by the inulin (Pourfarzad et al., 2015). The increase in O-H bond in the FTIR study was reflected in broad bands, indicating the bond of agave fructans with water, specifically the O-H linkages.

4. Conclusion

Knowing the temperatures at which important transitions take place is relevant information when predicting and controlling the optimal storage conditions required in ice cream, in order to preserve its characteristics and sensory properties, and to extend its shelf life. The use of agave fructans as a fat and sugar replacer influenced the thermal properties of ice cream. Samples with approximately 3.0 g/100 mL of agave fructans reduced the amount of ice and free water, and therefore increased bound water. Glass transition temperature was positively affected by formulations with higher agave fructans and moisture content in both treatments. Agave fructans have the ability to decrease the formation of ice crystals since they form a three-dimensional network that traps water, provoking a slow melt, which improves general quality. FTIR spectra showed an increase in the magnitudes of several bands, mainly in the O-H group that corresponds to polymeric hydrogen bonding caused by agave fructans and bound water. Finally, with this work we were able to validate that agave fructans (a fructose polymer similar to inulin but with different structural arrangements) improved some of the thermal properties that are affected by the reduction of fat and sugar in ice cream, making this ingredient a viable alternative as a replacer that does not provide calories. Nevertheless, for the next step it is suggested that a detailed sensory characterization be carried out to complement our results at this stage.

Acknowledgements

The authors are grateful for the financial support of DGAPA PAPIIT (project IT201417). A. Pintor-Jardines thanks CONACYT (México) for her Ph.D. scholarship in the Biotechnology Postgraduate Program (grant 246848). H. Escalona-Buendia belongs to the AGARED reasearch network sponsored by CONACYT (No. 281618).

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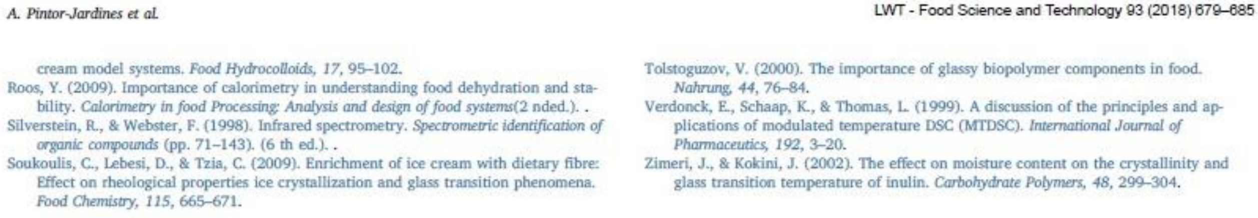

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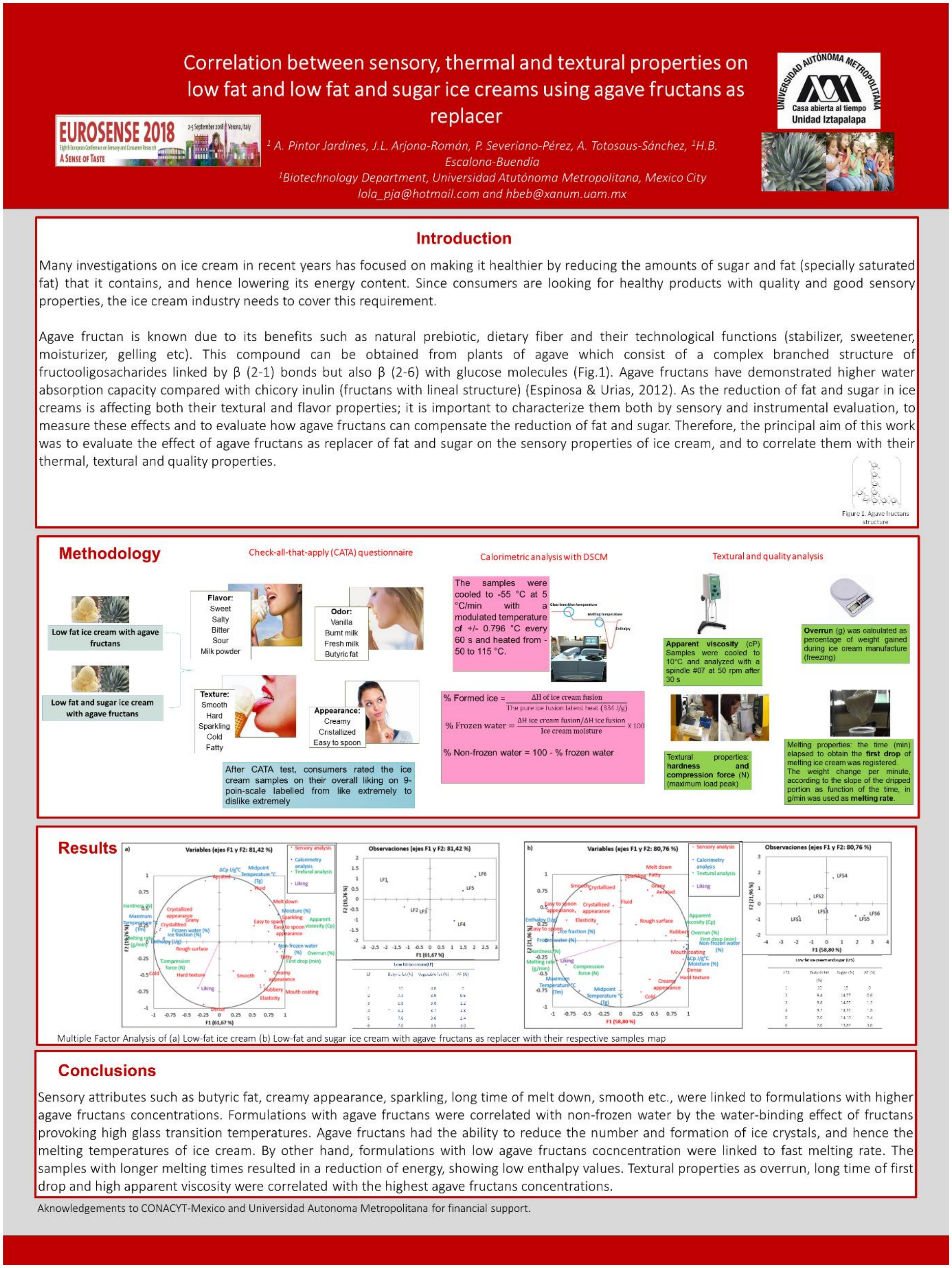
6. Artículo 3- Agave fructans as fat and sugar replacers in ice cream: sensory, thermal and textural properties.

Introducción

A lo largo de este capítulo se explican los resultados obtenidos de los análisis de las propiedades sensoriales, térmicas y de textura, así como su correlación. Este estudio se le realizó a helados reducidos en grasa y helados reducidos en grasa y azúcar, usando fructanos de agave (0-3%) como remplazo. Las formulaciones con menos de 1.2% de fructanos de agave provocaron atributos sensoriales “indeseables” relacionados con la cantidad y el tamaño de los cristales de hielo, como la textura y apariencia cristalizada y granulada, sensación de frío, etc., que a su vez se relacionaron con propiedades térmicas como agua congelada, fracción de hielo y valores de entalpías altas. Estos resultados fueron correlacionados con propiedades de textura como altos valores de dureza y cortos tiempos de derretimiento.

Por el contrario, las muestras que contenían concentraciones de agave entre 1.2 y 3.0% mostraron helados con características “más deseables” como fueron largos tiempos de derretimiento, textura suave, cremosa, fluida y una sensación a grasa. Estas propiedades se relacionaron directamente con bajas concentraciones de agua no congelada, altas temperaturas de transición vítrea, texturas suaves, y altos valores de viscosidad y overrun. En cuanto al agrado, todas las muestras estaban en la zona de agrado, pero los evaluadores prefieren las muestras que contienen todos los compuestos.



Correlation between sensory, thermal and textural properties on low fat and low fat and sugar ice creams using agave fructans as replacer

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Introduction

Many investigations on ice cream in recent years has focused on making it healthier by reducing the amounts of sugar and fat (specially saturated fat) that it contains, and hence lowering its energy content. Since consumers are looking for healthy products with quality and good sensory properties, the ice cream industry needs to cover this requirement.

Agave fructan is known due to its benefits such as natural prebiotic, dietary fiber and their technological functions (stabilizer, sweetener, moisturizer, gelling etc). This compound can be obtained from plants of agave which consist of a complex branched structure of fructooligosacharides linked by β (2-1) bonds but also β (2-6) with glucose molecules (Fig.1). Agave fructans have demonstrated higher water absorption capacity compared with chicory inulin (fructans with lineal structure) (Espinosa & Urias, 2012). As the reduction of fat and sugar in ice creams is affecting both their textural and flavor properties; it is important to characterize them both by sensory and instrumental evaluation, to measure these effects and to evaluate how agave fructans can compensate the reduction of fat and sugar. Therefore, the principal aim of this work was to evaluate the effect of agave fructans as replacer of fat and sugar on the sensory properties of ice cream, and to correlate them with their thermal, textural and quality properties.

Figure 1. Agave fructans structure

Methodology

Low fat ice cream with agave fructans

Low fat and sugar ice cream with agave fructans

Check-all-that-apply (CATA) questionnaire

Flavor:
Sweet
Salty
Bitter
Sour
Milk powder

Odor:
Vanilla
Burnt milk
Fresh milk
Butyric fat

Texture:
Smooth
Hard
Sparkling
Cold
Fatty

Appearance:
Creamy
Cristallized
Easy to spoon

After CATA test, consumers rated the ice cream samples on their overall liking on 9-poin-scale labelled from like extremely to dislike extremely

Calorimetric analysis with DSCM

The samples were cooled to -55 °C at 5 °C/min with a modulated temperature of +/- 0.796 °C every 60 s and heated from -50 to 115 °C.

$$\% \text{ Formed ice} = \frac{\Delta H \text{ of ice cream fusion}}{\text{The pure ice fusion latent heat (334 J/g)}}$$

$$\% \text{ Frozen water} = \frac{\Delta H \text{ ice cream fusion}/\Delta H \text{ ice fusion}}{\text{Ice cream moisture}} \times 100$$

% Non-frozen water = 100 - % frozen water

Textural and quality analysis

Apparent viscosity (cP) Samples were cooled to 10°C and analyzed with a spindle #07 at 50 rpm after 30 s

Overrun (g) was calculated as percentage of weight gained during ice cream manufacture (freezing)

Textural properties: **hardness** and **compression force** (N) (maximum load peak)

Melting properties: the time (min) elapsed to obtain the **first drop** of melting ice cream was registered. The weight change per minute, according to the slope of the dripped portion as function of the time, in g/min was used as **melting rate**.

Results

Multiple Factor Analysis of (a) Low-fat ice cream (b) Low-fat and sugar ice cream with agave fructans as replacer with their respective samples map

Conclusions

Sensory attributes such as butyric fat, creamy appearance, sparkling, long time of melt down, smooth etc., were linked to formulations with higher agave fructans concentrations. Formulations with agave fructans were correlated with non-frozen water by the water-binding effect of fructans provoking high glass transition temperatures. Agave fructans had the ability to reduce the number and formation of ice crystals, and hence the melting temperatures of ice cream. By other hand, formulations with low agave fructans cocncentration were linked to fast melting rate. The samples with longer melting times resulted in a reduction of energy, showing low enthalpy values. Textural properties as overrun, long time of first drop and high apparent viscosity were correlated with the highest agave fructans concentrations.

Aknowledgements to CONACYT-Mexico and Universidad Autonoma Metropolitana for financial support.

Figura 14.Congreso internacional, Eurosense, "Correlation between sensory, thermal and textural properties on low fat and low fat and sugar ice cream using agave fructans as replacer.

Agave fructans as fat and sugar replacers in ice cream: sensory, thermal and textural properties (Food Hydrocolloids)

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Abstract

The principal aim of this work was to investigate the feasibility of replacing fat and sugar with agave fructans to produce both low-fat, and low-fat and sugar ice cream formulations. For this purpose, agave fructans (0 to 3.0%) were added to ice cream formulations to explore relationships between their sensory, thermal and textural properties. Formulations with less than 1.2 % of agave fructans provoked sensory attributes linked to the amount and size of ice crystals, such as crystallized (texture and appearance), grainy and cold sensation. These were related to thermal properties such as frozen water, ice fraction, enthalpy and maximum temperature, as well as to textural properties such as hardness, melting rate and compression force.

On the other hand, the samples with concentrations between 1.2 to 3.0% of agave fructans showed ice cream formulations with long melting times; smooth, creamy and fluid textures, the same as a fatty sensation. These properties were directly related to low non-frozen water concentrations, high glass transition temperatures

and ΔCp values, as well as textural properties, such as apparent viscosity and overrun. After an additional hedonic test, even though all the samples showed an average liking score, consumers preferred the samples with higher amounts of fat and sugar.

1. Introduction

Many investigations into ice cream in recent years have focused on lowering its energy content by reducing the amounts of sugar and fat (especially saturated fat). Since consumers are looking for healthy products with quality and good sensory properties, the ice cream industry needs to cover this requirement. Agave fructans are known for their benefits, such as natural prebiotic, dietary fiber, and their technological functions (stabilizer, sweetener, moisturizer, gelling etc.). They can be obtained from agave plants and consist of a complex branched structure of fructooligosacharides linked by β (2-1) and β (2-6) bonds, also with glucose molecules. Agave fructans have demonstrated a higher water absorption capacity compared with chicory inulin (fructans with lineal structure) (Espinosa & Urias, 2012).

Ingredients and processing conditions, especially during periods of temperature fluctuations, are important in ice cream manufacture since they determine its sensory perception and texture. The sensory properties are food characteristics detected by the senses of sight, smell, taste, touch and hearing, such as flavor, texture and appearance. For ice cream, the perception of flavors can be affected because they are perceived less intensely at low temperatures; for this reason, ice cream and sorbets are more strongly flavored than products consumed at warmer temperatures. Texture is another important sensory parameter that is the response of the tactile senses to physical stimuli resulting from contact between some part of the body and the food. The tactile sense (touch) is the primary method for sensing texture, but kinesthetics (sense of movement and position) and sometimes sight (degree of slump, rate of flow), and sound (associated with crisp, crunchy and crackly textures) are also used to evaluate texture (Bourne, 2002). Many food companies have used a descriptive profile to provide a qualitative and quantitative representation of aspects that are perceived by humans, enabling measurement of a sensory reaction to the stimuli from the use of a product. All descriptive analyses implicate the detection (discrimination) and the description of both qualitative and quantitative sensory aspects of a product that are relevant to consumers (Meilgaard et al., 2007), but using trained assessors for the actual evaluation. Nevertheless, a popular alternative to the fast-descriptive sensory methods is Check All That Apply (CATA), which has been used as a bridge between different areas of research, product development, and consumer science, resulting in a link between a product's characteristics and consumer perception (Varela and Ares, 2012).

The texture and stability of ice creams can be explained by the thermal properties. They are important for understanding the freezing time, just as for simulating the temperature field variations throughout the frozen material during the freezing and storage periods. Achieving a control of thermal properties improves the quality and the stability of the frozen foods, in this case of the ice creams. These properties have been studied to simulate the freezing process, which has a great importance in the design of equipment and in the control of their operating costs (Cogné at al., 2003). Moreover, the effect of sugar and fat reduction in ice formation has already been studied by using the thermal properties of ice creams (Pintor et al., 2018). On the other hand, instrumental control of ice cream texture and quality is commonly performed by the analyses of several properties, such as viscosity of the base (mix before freezing), overrun, hardness and compression force, first drop and melting rate. All of them have been previously used to characterize the structure of ice cream and to optimize new formulations (Pintor and Totosaus, 2012; Pintor et al., 2013; 2017).

As the reduction of fat and sugar in ice creams affects both their textural and flavor properties, it is important to characterize them both by sensory and instrumental evaluation in order to determine these effects and, the focus of this study, to understand how agave fructans could compensate for the reduction of fat and sugar. Therefore, the principal aim of this work was to evaluate the effect of agave fructans as a replacer for fat and sugar on the sensory properties of ice cream, and to correlate them with their thermal, textural and quality properties.

2. Materials and methods

2.1. Ice cream base manufacture

Six low fat (LF), and six low fat and low sugar (LFS) formulations were prepared according to previous investigations (Pintor et al., 2013 and Pintor et al., 2017; respectively). Five LF and five LFS formulations were optimized (Table 1) with agave fructans which were processed as follows: The ingredients, such as non-fat dry milk and whey protein concentrate (8.0% and 4.0%, respectively, DILAC S.A. de C.V., Cuautitlan Izcalli, Mexico), emulsifier (sorbitan monoestereate and diglycerides, 0.25%, ARCY S.A. de C.V., Ecatepec, Mexico), agave fructans (0.0-3.0%, Vaserco, S.R.L. de C.V., Guadalajara, Mexico, as a fat and sugar replacer) which were characterized by Size-Exclusion Chromatograph, polymerization degree (DP) was 17.1 ± 0.2, having a proportion of fructans (DP > 10) at 72.5g/100g ± 0.2 and FOS (DP 3 -10) of 27.4g/100g ± 0.2, with residual sucrose (0.48g/100g), glucose (1.88g/100g) and fructose (4.94g/100g) contents. In the same way, anhydrous butyric fat (7.0-10%, ARCY S.A. de C.V., Ecatepec, Mexico), vegetable fat (3.5-4.0%, La Mixteca, Ecatepec, Mexico), were mixed in water at 60°C using a Stick mixer Oster-2609 (Oater, Boca Raton, Florida). All ice cream samples were pasteurized at 70°C for 30 min and then cooled in a bath at 4°C for 24 h. The base mixes were frozen in a frozen ice-cream CIM-50RSA machine (Cuisinart, East Windors) for 25 minutes, before being hardened at -25°C for 24 h.

Table 1. Ice cream formulations.

Low fat ice cream (LF)				Low fat and low sugar ice cream (LFS)			
Treatment	Butyric fat (%)	Vegetable fat (%)	Agave fructans (%)	Treatment	Butyric fat (%)	Sugar (%)	Agave fructans (%)
LF1	10	4.0	0	LFS1	10	15	0
LF2	9.4	3.9	0.6	LFS2	9.4	14.77	0.6
LF3	8.8	3.8	1.2	LFS3	8.8	14.55	1.2
LF4	8.2	3.7	1.8	LFS4	8.2	14.32	1.8
LF5	7.6	3.6	2.4	LFS5	7.6	14.10	2.4
LF6	7.0	3.5	3.0	LFS6	7.0	13.2	3.0

2.2. Sensory evaluation

For sensory evaluation, seventy-two habitual, untrained ice cream consumers were recruited to assess twelve different ice cream formulations (Table 1). The evaluation was performed in a standardized room equipped with individual booths (ISO, 2007).

The samples were coded with 3-digit numbers according to a random design given by Fizz Software version 2.50 (Biosystems, Counternon, France). All samples were served at -23°C in individual plastic containers of 35 ml capacity. The first session was conducted to explain the CATA methodology and the consumers described six random samples of ice cream. In the second session the other six samples were evaluated.

2.2.1. Liking test

The consumers tasted the ice cream samples and rated their overall liking on a 9-point-scale labelled from “like extremely” to “dislike extremely”.

2.2.2. Check-all-that-apply (CATA) questionnaire

In order to avoid saturation, the CATA test was carried out in two parts; first for the six low-fat (LF) samples, and the second for the six low-fat and low sugar (LFS) ice cream formulations. Some attributes were selected from a panel specifically trained for ice cream description (Varela et al. 2014). Additionally, with the purpose of complementing this list of attributes, a preliminary CATA session was carried out, asking consumers to both verify the vocabulary and to include some terms that were not on the list. The sum of all attributes was considered in the formal CATA questionnaire, and they were divided into four groups, *appearance*: creamy, crystallized and easy to spoon; *odor*: vanilla, burnt milk, butyric fat and fresh milk; *flavor*: sweet, salty, sour, bitter, milk powder, caramel and metallic; and *texture*: smooth, hard, sparkling, gummy, cold, fatty, dense, sandy, fluid, easy to spoon, crystallized, melt down, mouth coating, elasticity, grainy and rough surface. For each sample, the consumers selected the appropriate attributes that described it.

2.3. Modulated Differential Scanner Calorimetry (MDSC)

The thermal properties were measured in a previous study (Pintor et al., 2018), obtaining the ice fraction, frozen water, non-frozen water percentage, glass transition °C (Tg), ΔCp, fusion temperature °C (Tm) and enthalpy (J/g). MDSC was used to analyze the thermal properties of the ice cream samples (Table1), obtaining heating and cooling curves with a TA Instrument Universal Analysis 2000 V 4.5A software. The method was modified from Blond methodology (1994) as follows: cooling to -55 °C at 5 °C/min with a modulated temperature of +/- 0.796 °C every 60 seconds and heating from -50 to 115 °C.

2.4. Textural and quality analysis

All samples were analyzed as described by Pintor et al., 2017. The ice cream bases, which are the products before being frozen, were measured for apparent viscosity (cP). The ice cream yield, better known as overrun, was calculated as a percentage of weight gained during ice cream manufacture (freezing). Melting properties such as first drop (min), melting rate (g/min) and textural properties such as hardness and compression force (N), were also determined.

2.5. Data analysis

The CATA questionnaire and a liking test were designed using the Fizz Software version 2.50 (Biosystems, Counternon, France). The overall liking data was used as illustrative data in the analysis. Analysis of variance (one-way ANOVA) was applied to the consumer liking scores. The results obtained from CATA were analyzed by Factorial Correspondence Analysis (FCA). Thermal and textural data analysis was carried out by Principal Components Analysis (PCA). Finally, Multiple Factor Analysis (MFA) was used in order to obtain a bi-dimensional representation of the samples and the correlation between sensory, thermal and textural analysis. All statistics were performed using XLStat 2014 software (Addinsoft, Paris, France).

3. Results and discussions

The key point of this research was to evaluate the behavior of the different formulation groups by means of the correlation between their sensorial, thermal and textural properties. Nevertheless, it explores the sensory study in greater depth, since thermal and textural analysis have already been analyzed and discussed in previous investigations (Pintor et al., 2017; 2018).

3.1. Overall liking results and CATA methodology

The CATA test was to gain a better understanding of which sensory descriptors defined the ice cream samples and were responsible for the hedonic response of consumers. There were significant differences between samples ($P<0.05$) according to the independence Chi-Square Test, and the relation between samples and descriptor frequencies are shown on the FCA maps (Figs 1 and 2). Based on results from CATA of low-fat ice cream formulations with agave fructans as the replacer (Fig. 1), LF-1 and LF-2 were closely correlated with metallic and bitter flavor; fresh milk odor; crystallized (appearance and texture) grainy, sandy, rough surface, cold and hard texture. These are two very similar formulations with higher fat and lower agave fructans content. The samples LF-3 to LF-5 were related to sweet, salty, milk powder and caramel flavor; vanilla, burnt milk, butyric fat odor; gummy, fatty, elasticity, mouth coating and dense texture; and creamy appearance. Finally, the formulation LF-6 (with higher agave fructans and lower fat content) was very close to the control sample (with full fat content) and had the most desired attributes for ice cream. Sour flavor (could be the only unwanted attribute); fluid, long melting time , sparkling texture and easy to spoon (both for appearance and texture) were correlated with LF-6 ice cream. The samples LF-3 and LF-4 had significantly higher values for overall liking (Table 2).

For low fat and low sugar ice cream formulations with agave fructans as the replacer (Fig. 2), LFS-1 and LFS-2 (samples with higher sugar and fat content) were related to sweet flavor; fresh milk, vanilla odor; cold, hard texture and easy to spoon appearance and texture. The samples LFS-3 and LFS-4 were close and described as caramel flavor and smooth, long melting time, sparkling, fluid, elasticity textures. The formulation LFS-5 was described as salty and burnt milk flavor; butyric fat odor; rough surface crystallized appearance and texture. For LFS-6 the consumers chose attributes such as metallic, sour, bitter, powder milk flavors, mouth coating, dense, grainy, sandy, gummy and fatty; creamy appearance. In spite of a reduction of fat and sugar in LFS-4, 5, and 6, agave fructans had the ability to compensate for these compounds, since it was possible to identify positive attributes related to a high-quality ice cream.

According to mean liking scores (Table 2), there are significant differences in LF-2, 5 and 6 against the control and LFS-4, 5 and 6 against the control. In general, all samples had a medium liking value (around 6 points on a 9-point hedonic scale). Nevertheless, the samples that the assessors liked most were those that contained all the compounds (fat and sugar). Probably, in ice cream reduced in fat and sugar, the agave fructans had the ability to compensate for texture but not taste. Another possible cause could be that the evaluation of all ice cream samples was carried out without any added flavor; therefore, it is possible that the incorporation of a well-known flavor, such as vanilla, would improve both the consumers' perception and liking scores.

Table 2. Mean liking score for the different ice cream formulations on a 9-point hedonic scale for low-fat (LF) and low- fat and sugar (LFS) ice cream.

Sample	Overall liking	Sample	Overall liking
LF-3	6.5^{a}	LFS-1	6.5^{a}
LF-4	6.4^{ab}	LFS-2	6.3^{ab}
Control	6.2^{bc}	Control	6.2^{bc}
LF-1	6.2^{bc}	LFS-3	6.0^{cd}
LF-2	6.0^{cd}	LFS-5	5.9^{de}
LF-5	5.9^{de}	LFS-4	5.8^{ef}
LF-6	5.7^{e}	LFS-6	5.7^{f}

$^{a-f}$Mean scores with the same letter in the same column are not significantly different (P<0.05)

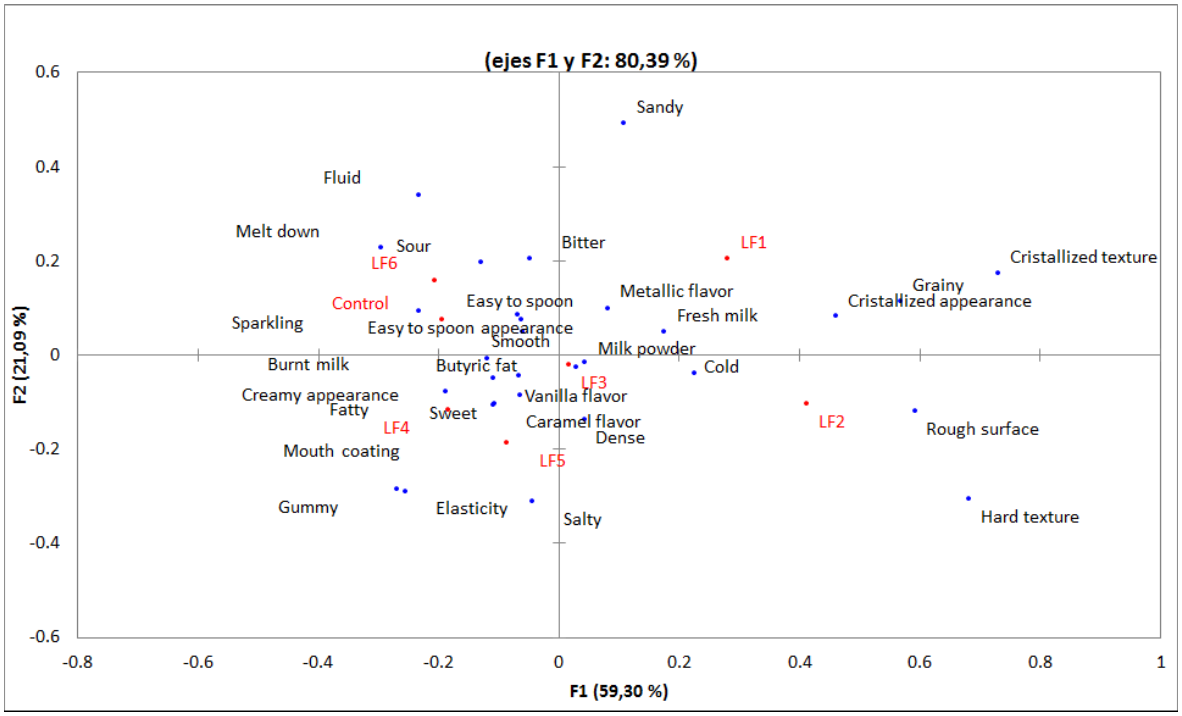

Figure 1. Factorial Correspondence Analysis (FCA) of sensory attributes and samples of Low fat with agave fructans ice cream.

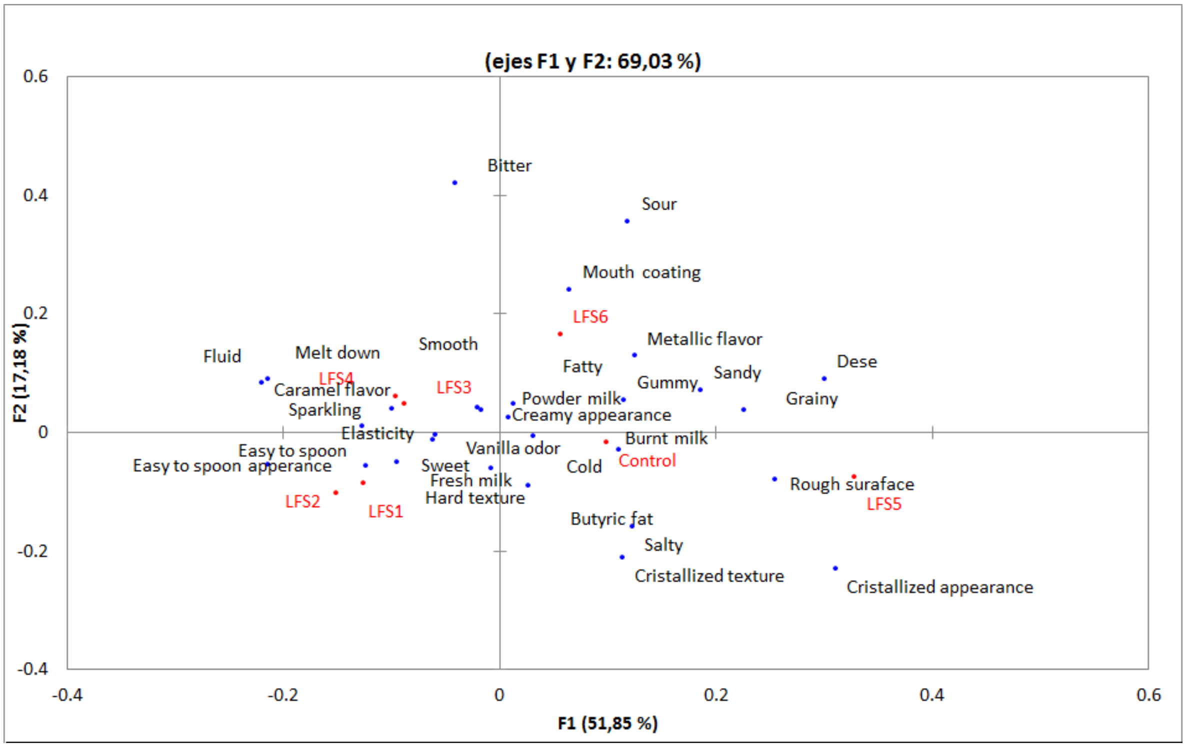

Figure 2. Factorial Correspondence Analysis (FCA) of sensory attributes and samples of low fat and low sugar with agave fructans ice cream.

3.1.1. Sensation of odor and flavor.

The quality of ice cream depends on several factors that define the sensory attributes of ice cream as sweet, flavor, body and texture and cold sensation, as perceived by consumers. Flavor is a complex combination of odors, smelt through the nose (*i.e.* volatile compounds), and taste, experienced by the tongue (*i.e.* non-volatile liquids or solids). Odors are distinguished via olfactory nerve endings in the upper part on the nose (Alvarez, 2009). The change of serum phase composition, viscosity and increase in ice crystals seem to play an essential role in the release of flavor. As freezing continues in ice cream, the unfrozen phase becomes more and more concentrated and the temperature continues to decrease. This leads to an increase in viscosity in the unfrozen phase, until the viscosity is sufficiently high that the concentrated unfrozen phase becomes glassy. During consumption of ice cream, different intensities of flavors are perceived as the temperature increases in the mouth. Due to the fact that the low temperature of ice cream induces numbing of the tongue, more time is required to perceive and evaluate the flavor. The large amount of frozen water caused by the recrystallization phenomena, causes difficulties in the release of volatile flavor compounds, as ice cream is diluted by melting ice crystals and saliva. All these phenomena are affecting flavor perception and intensity. Thus, any flavor present in ice cream will become more apparent as the sample warms up, which occurs in the mouth as well as on the sample plate (Alvarez, 2009). On the other hand, there are studies showing that fat controls the release of flavor volatiles during the consumption of ice cream. For example, the maximum intensity of cherry flavor increases significantly as the fat levels of ice cream decrease (Chung and Grün, 2003).

According to our results for LF ice cream (Fig. 1), the attribute sweet was chosen in samples with intermediate agave fructans content, although the sugar concentration was the same for all formulations. In LFS ice cream formulations (Fig. 2), sweet was perceived in samples with the highest sugar content, as expected. Not much is known about how much sweetness agave fructans confer. In general, oligofructose in the pure form has a sweetness of about 35% in comparison with sucrose; however, depending on several factors they could have some free fructose that is providing some degree of sweetness For LF ice cream samples, sweet flavor was close to caramel flavor and vanilla, burnt milk and butyric fat odor. In another work, similar behavior was observed in which sweetness ratings increased with sugar content and higher vanilla, almond, buttery, custard/eggy, sweetness, fatty, creamy, doughy and mouth coating characteristics were found (Guinard et al., 1997). Sour, bitter, salty and metallic flavors were distinguished by consumers in LF and LFS ice cream formulations (Figs. 1 and 2). Salty flavors may arise from the non-fat milk solids, especially if whey powder is used, due to the higher quantity of natural milk salts. These attributes are considered as defects in frozen dairy desserts and they are caused by the high milk and whey powder content. Nevertheless, these off-flavors are suppressed by sweetness (Alvarez, 2009). According to odor characteristics, vanilla, butyric fat, burnt milk and fresh milk odor perhaps came from dairy products and fat.

3.1.2. Texture and appearance

The appearance and texture of products are sometimes the most important features for consumers when they choose a food, which is possibly a fact for ice creams. Texture is the response of the tactile sense to physical stimuli and results from contact between some part of the body and food. The consumption of the products also depends on appearance, which includes color, size, shape and surface texture (Meilgaard et al., 2007). In ice cream, the mechanical forces imparted by the tongue, the upper palate and the teeth will determine the overall perception of the texture (Aime et al., 2001).

3.2. Thermal and textural properties

The thermal results have been reported and discussed in depth in Pintor et al. (2018). Basically, they were evaluated by Modulated Differential Scanning Calorimetry and approximately 3.0% of agave fructans were used in low fat, and low fat and sugar ice cream formulations, to replace fat and/or sugar. Those results were subjected to Principal Component Analysis (Figures 3(a) and 4(a) respectively). For both groups of formulations (LF and LFS), the samples that contained lower concentrations of agave fructans (less than 1.2%) were correlated with frozen water (%), ice fraction (%), maximum temperature (°C) and enthalpy (J/g). The samples with concentrations near to 3.0% were related to transition glass (°C), ΔCp (J/g °C), moisture and non-frozen water (%).

Textural analysis was carried out, also based on a previous study (Pintor et al., 2017). Results were used to explore the relation of the textural properties (Tables 3 and 4) with each formulation, also using Principal Component Analysis. PCA showed (Figures 3(b) for LF and 4 (b) for LFS) how the samples with a lower amount of agave fructans (less than 0.6%) were correlated with textural properties such as compression force (N), hardness (N) and melting rate (g/min), while samples with approximately 1.8% of agave fructans were related to overrun (%) and first drop. Apparent viscosity was associated with a high agave fructans concentration (approximately 3.0%). There is a deeper discussion below when explaining the correlation with sensory and thermal properties.

Table 3. Texture properties results of low fat ice cream using agave fructans as replacer

Sample	Apparent viscosity (Cp)	Overrun (%)	Melting rate (g/min)	First drop (min)	Hardness (N)	Compression force (N)
LF 1	2600^{a}	19^{a}	1.2^{d}	20^{a}	152.9^{f}	148.6^{d}
LF 2	2730^{ab}	20^{ab}	0.9^{c}	32^{b}	88.7d^{e}	126.7^{cd}
LF 3	3050^{c}	35^{c}	0.5^{bc}	53^{c}	68.3^{d}	120.4^{cd}
LF 4	3800^{cd}	37^{cd}	0.3^{ab}	60^{d}	41.0^{c}	122.8^{c}
LF 5	4320de	37^{cd}	0.3^{ab}	65de	16.0^{ab}	97.9^{ab}
LF 6	4430^{ef}	40^{d}	0.1^{a}	67de	7.7^{a}	81.9^{a}

$^{a-f}$*Mean scores with the same letter in the same column are not significantly different (P<0.05)*

Table 4. Texture properties results of low fat and sugar ice cream using agave fructans as replacer

Sample	Apparent viscosity (Cp)	Overrun (%)	Melting rate (g/min)	First drop (min)	Hardness (N)	Compression force (N)
LFS 1	2720^{a}	16^{a}	1.1^{c}	18^{a}	160.8^{ef}	290.5^{c}
LFS 2	3000^{ab}	19^{ab}	0.8^{bc}	25^{b}	147.9^{e}	136.9^{bc}
LFS 3	3080^{ab}	24^{ab}	0.7^{bc}	37^{bc}	85.2^{d}	126.9^{bc}
LFS 4	3960^{c}	32^{c}	0.4^{ab}	40^{d}	67.3^{c}	108.7^{bc}
LFS 5	4200^{cd}	42^{cd}	0.3^{ab}	55^{e}	25.2^{ab}	101.7^{b}
LFS 6	4800^{cd}	44^{cd}	0.1^{a}	60^{f}	18.9^{a}	79.3^{a}

$^{a-f}$Mean scores with the same letter in the same column are not significantly different (P<0.05)

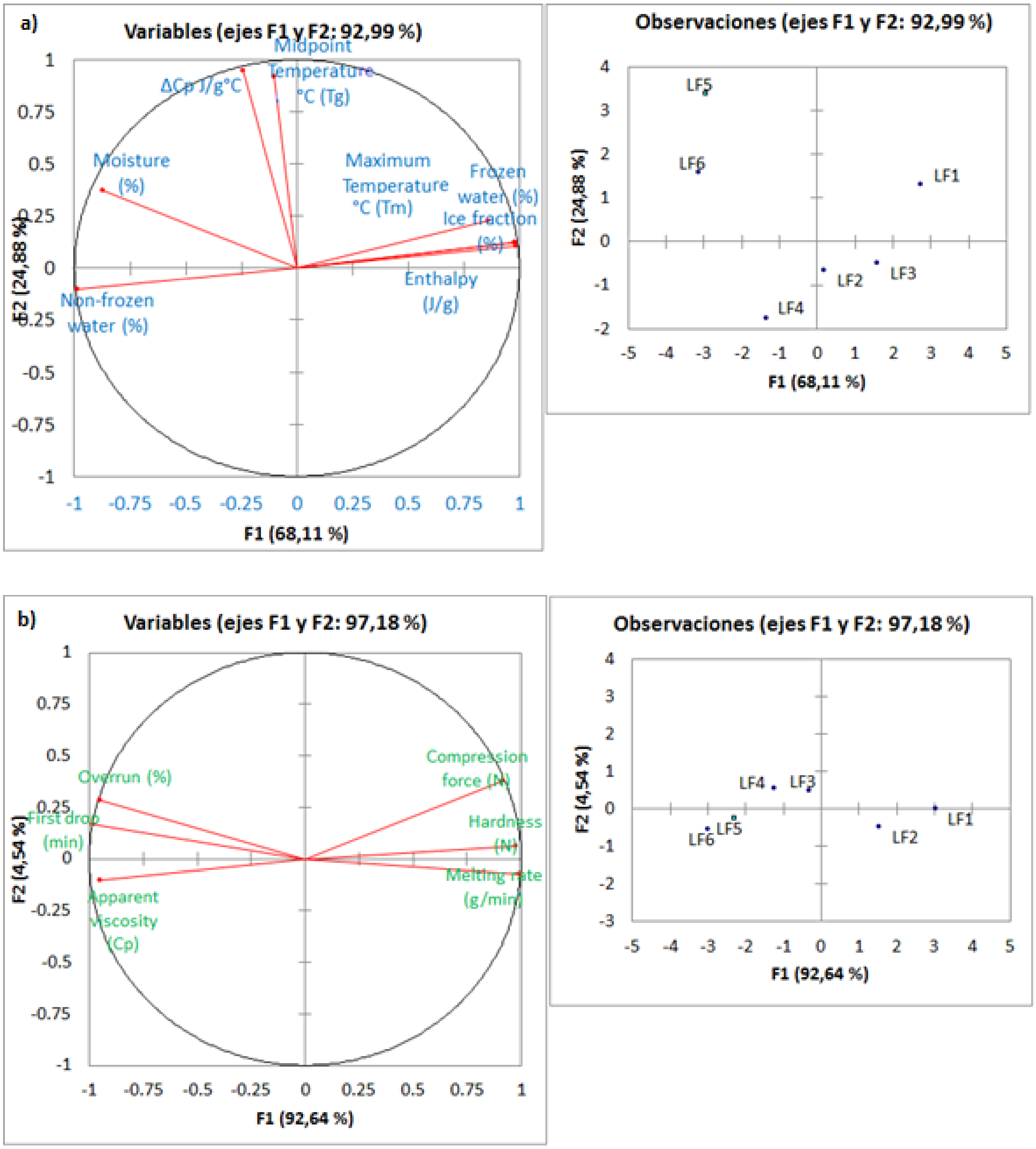

Figure 3. Principal Components Analysis of thermal (a) and textural properties (b) correlations of low fat with agave fructans ice cream (LF), where LF-1 had lower and LF-6 had higher agave fructans concentration.

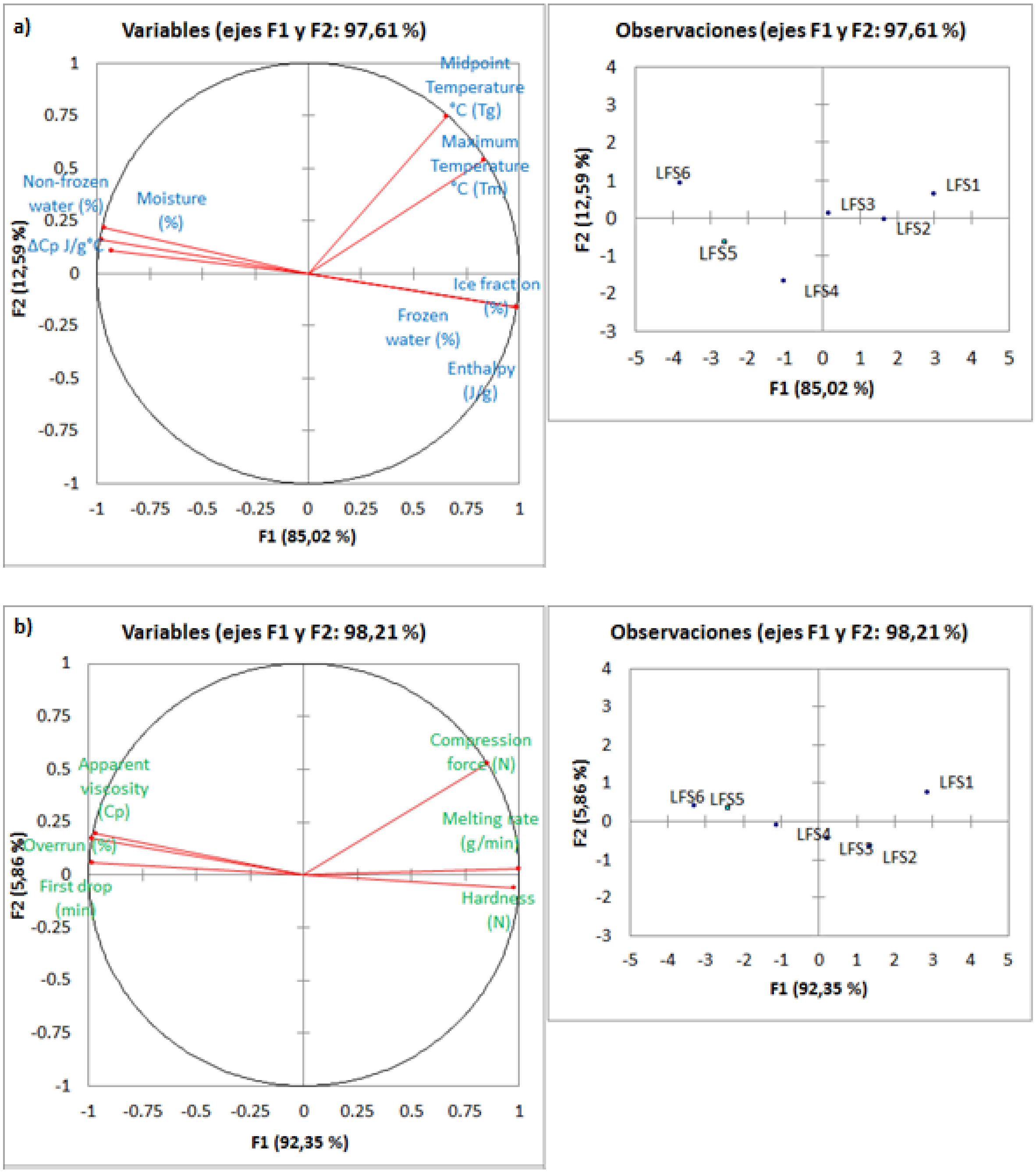

Figure 4. Principal Components Analysis of thermal (a) and textural properties (b) correlations of low fat and sugar with agave fructans ice cream (LFS), where LFS-1 had lower and LFS-6 had higher agave fructans concentration.

3.3. Correlation between sensory, thermal and textural properties

MFA made it possible to work with different groups of variables: sensory, thermal and textural, to obtain a concise representation of all the variables studied and to link them to samples positioning. Figures 5 (a and b) show the MFA for LF and LFS ice cream respectively, in which the two first factors of MFA explained 61.67 and 19.76% of variability of the data for LF and 58.80 and 21.96% for LFS. The RV coefficients, indicating the correlation between sensory, calorimetry and textural analysis, are observed in Tables 5 and 6.

For LF ice cream there was a good correlation between sensory and textural analysis (0.739). In the same way, textural and calorimetry analyses were well correlated with 0.741. Liking (used as a supplementary variable) did not have a good correlation. According to MFA analysis between sensory, calorimetry and textural analysis, the upper right quadrant shows that frozen water, ice fraction, enthalpy, melting temperature (thermal properties); hardness and melting rate (textural properties) were correlated positively with grainy, crystallized appearance and texture (sensory attributes). In the lower right quadrant, compression force was correlated with rough surface, hard texture and overall liking. The lower left quadrant indicated that only overrun and first drop were linked to fatty, smooth, rubbery, elasticity, creamy appearance and mouth coating. Finally, in the upper left quadrant ΔCp, glass transition temperature, moisture, non-frozen water and apparent viscosity were correlated with aerated, fluid, melting , sparkling, easy to spoon appearance and texture.

The RV coefficients (Table 6) for LFS show that calorimetry and textural analysis had a high correlation (0.985) with each other, and also had a high correlation with liking (0.913 and 0.928 respectively). According to MFA for LFS, the thermal parameters enthalpy and ice fraction were correlated with elasticity, crystallized appearance and texture, smooth, easy to spoon appearance and texture (upper right quadrant). In the lower right quadrant, we observed that melting temperature, frozen water, glass transition temperature, hardness, melting rate, compression force correlated strongly with overall liking. The parameters non-frozen water, ΔCp and moisture in the lower left quadrant were correlated positively with mouth coating, dense, hard texture, creamy appearance and cold. In the upper left quadrant, apparent viscosity, overrun, and first drop were linked to melting, fatty, grainy, sparkling, aerated, rough surface and rubbery.

Table 5. Coefficient RV between sensory, calorimetry, textural and liking analysis for LF ice cream formulations

	Sensory	Calorimetry	Textural	Liking	AFM
Sensory	1.000	0.574	**0.739**	0.240	0.873
Calorimetry	0.574	1.000	0.741	0.489	0.867
Textural	0.739	**0.741**	1.000	0.136	0.926
Liking	0.240	0.489	0.136	1.000	0.327
AFM	0.873	0.867	0.926	0.327	1.000

Table 6. Coefficient RV between sensory, calorimetry, textural and liking analysis for LFS ice cream formulations

Test	Sensory	Calorimetry	Texture	Liking	AFM
Sensory	1.000	0.660	0.571	0.502	0.849
Calorimetry	0.660	1.000	0.985	0.913	0.956
Texture	0.571	**0.985**	1.000	0.928	0.917
Liking	0.502	**0.913**	**0.928**	1.000	0.838
AFM	0.849	0.956	0.917	0.838	1.000

In general, the RV coefficients for liking were lower in LF ice cream formulations. On the other hand, for low sugar and fat (LFS) ice creams, there were higher RV coefficients when liking was correlated with calorimetry and texture properties (0.913 and 0.928, respectively). By decreasing the fat and sugar content in ice cream formulations, several attributes were affected for both ice cream groups (LF and LFS). The thermal properties such as frozen water, ice fraction, melt temperature (T´m) and enthalpy were in samples with lower agave fructans concentrations (0-1.2%) (upper and lower right quadrants). Maybe for these properties, the water was in a free state due to the absence of hydrophilic sites (provided by fructans) that bind water. It is known that only free water can undergo possible transitions in state such as ice crystallization (Cogné, Caillet, Andrieu, Laurent & Rivoire, 2003). The melt temperature (T´m) and enthalpy were placed in these quadrants; probably the large amount of free water led to melting first, using a greater amount of energy that was reflected in high enthalpy values. The higher amount of ice crystals, led to the textural properties such as hardness, compression force and melting rate. The ice crystals and ice phase volume contribute to ice cream hardness caused by free water freezing (Pintor et al., 2013). Consequently, a greater amount of free water was reflected in faster melting. In some papers it is clearly shown that during the melting process, the melting rate is highly dependent on the fat agglomerates in the unfrozen serum phase (Koxholt et al., 2011). Thermal and textural analysis were correlated with sensory attributes related to texture in the mouth, for example hard texture, rough surface, cold, crystallized appearance and texture, which were perceived due to the quantity and size of ice crystals.

The upper and lower left quadrants show that thermal properties such as non-frozen water, moisture, glass transition temperature (T´g) and ΔCp correlated well with the higher agave fructans concentration formulations, as well as different sensory attributes such as aerated, fluid, sparkling, easy to spoon, fatty, creamy appearance, smooth, rubbery, mouth coating and elasticity. In this case, the addition of agave fructans influenced the water freezing as the amount of free water was reduced. The moisture was affected because the fat and sugar were reduced, and it was replaced with water and agave fructans. The addition of agave fructans helped to increase glass temperatures which indicated that ice cream started to change from a viscoelastic liquid (rubber) to an amorphous solid (glass) with increased viscosity. This increase in temperature is related to the restriction of the mobility of water molecules and the thermodynamic stability caused by agave fructans. Glass transition implies a change in heat capacity (Cp) as moisture and agave fructans increase. In other investigations, chicory inulin helped to decrease crystallization in low fat ice cream. Crystallization was strongly dependent on the percentage of bound water, caused by both the influence of soluble solids in the aqueous phase and by the water-binding effect of fructans, which formed a gel-like network (El- Nagar, Clowes, Tudorica, Kuri & Brennan, 2002; Soukoulis, Lebesi & Tzia, 2009). Therefore, the addition of fructans has been used in ice cream to reduce fat and sugar due to its capacity to form aggregates that trap free water, reducing the ice crystal content formed during freezing, which is reflected in textural and sensorial properties (Akalin & Erisir, 2008; Akin & Kimaci, 2007; Ismail, Saleh-Al, & Metwalli, 2013; Meyer, Bayarri, Tarrega & Costell 2011; Pintor, Severiano & Totosaus, 2013).

These higher water and lower agave fructans samples were characterized with melting properties; this means that a higher free water concentration results in ice cream that melts faster when exposed to warm temperatures. Therefore, the use of agave fructans in samples with reduced fat and sugar helped to stabilize the ice cream, and decreasedmelting times.. In other studies, chicory inulin improved ice cream melting properties by creating a three-dimensional network that traps water, provoking a decrease in melting time (Akalin & Erisir, 2008; Karaca, Güven, Yaser, Kaya, & Kahyaoglu, 2009). Another parameter that correlated with these samples was enthalpy. The samples with higher free water resulted in an increase of enthalpy values. This means that more energy was needed to melt these samples (Soukoulis, Lebesi & Tzia, 2009; Zimeri & Konini, 2002).

Textural properties such as apparent viscosity, overrun and first drop, were correlated with each other. Some fat and sugar replacers, such as inulin, have been employed to increase viscosity in reduced fat ice cream (Aime et al., 2001; Aykan et al., 2008; Karaca et al., 2009). The increase in the apparent viscosity can be explained by the interactions of inulin and the liquid components of the ice cream mix. This effect is caused by both the contribution of soluble solids to the aqueous phase and by the water-binding effect of fructans, which form a gel-like network that modifies the viscosity mix (El-Nagar et al., 2002; Akin et al., 2007; Soukoulis et al., 2009). Melting properties, such as first drop, are inversely proportional to overrun, with a lower melting rate producing higher overrun values and vice versa (Sakurai et al., 1996; El-Nagar et al., 2002; Muse and Hartel, 2004; Sofjan and Hartel, 2004;

Akalin and Eris¸ir, 2008). In turn, higher overrun values result in slower melting, since air cells act as an insulator medium (Sakurai et al., 1996; Caillet et al., 2003; Marshall et al., 2003; Akalin and Eris¸ir, 2008). These thermal and textural properties correlated well with some sensory attributes which are desirable in a commercial ice cream (long melting times, creamy appearance, smooth, etc) and were perceived by consumers.

Thermal and textural properties are principally influenced by ice formation. The quantity and size of ice crystals during freezing and storage determine the hardness of ice cream, provoking cold sensation, crystallized and hard ice cream textures (Goff, 1997). Instrumental texture tests serve to determine the uniformity of the ice cream structure. Hardness is a penetration measure which indicates the force required to break the structure of ice crystals, air bubbles and emulsified fat globules. Force compression test is another way to know about ice cream structure. It simulates the deformation that occurs during mastication between the palate and the tongue (Clarke, 2004). In our study, the samples with agave fructans content showed lower compression force and hardness, and that was expected because fat replacers (such as water binding agents) reduced the hardness of ice cream causing smoother product (Roland et al., 1999; Karaca et al., 2009). Ice cream hardness is inversely related to fat and solid contents (Guinard et al., 1997; Roland et al., 1999; El- Nagar et al., 2002). Melting rate is another textural parameter that was related with the samples with lower agave fructans and this means that these samples melted faster than the rest of the ice cream. This textural parameter correlated with crystallized appearance and texture, grainy, rough surface and hard texture, which in turn were related to free water and a greater number of ice crystals, and therefore, greater melting rates. The use of agave fructans as a fat and sugar replacer resulted in longer first drop times, probably due to the fact that the fructans had the ability to immobilize large amounts of water. Other studies have used inulin to improve ice cream melting properties, since in non-fat and fat-reduced ice creams formulated with inulin the melting rate was lower; this could be due to the ability of inulin to retain water (Aykan et al., 2008; Akalin and Eris¸ir, 2008; Karaca et al., 2009). Samples 3 and 4 (for LF and LFS ice cream formulations) were related to overrun, which is the amount of air incorporated during the manufacture of ice cream. This parameter is associated to viscosity, since air bubbles act as an insulating medium and avoid rapid heat transfer from the medium to the ice crystals (Marshall et al., 2003). In other studies, high overrun was related to higher viscosities that promote air incorporation and the formation of smaller air cells (Akin et al., 2007; Chang and Hartel, 2002). In addition, the use of inulin caused high viscosities and therefore high overrun values (Akalin et al., 2008). According to our study, the samples with a higher agave fructans concentration correlated with viscosity as well as rubbery, sparkling and aerated sensory parameters, which are related with the amount of incorporated air and the viscosity of the samples. Probably, agave fructans had the capability to form small aggregates that trap water and increase viscosity. In several investigations about ice cream, inulin has been used as a fat and sugar replacer to improve viscosity in the base (Karaca et al., 2009; Akalin and Erişir, 2008; Akalin et al., 2007).

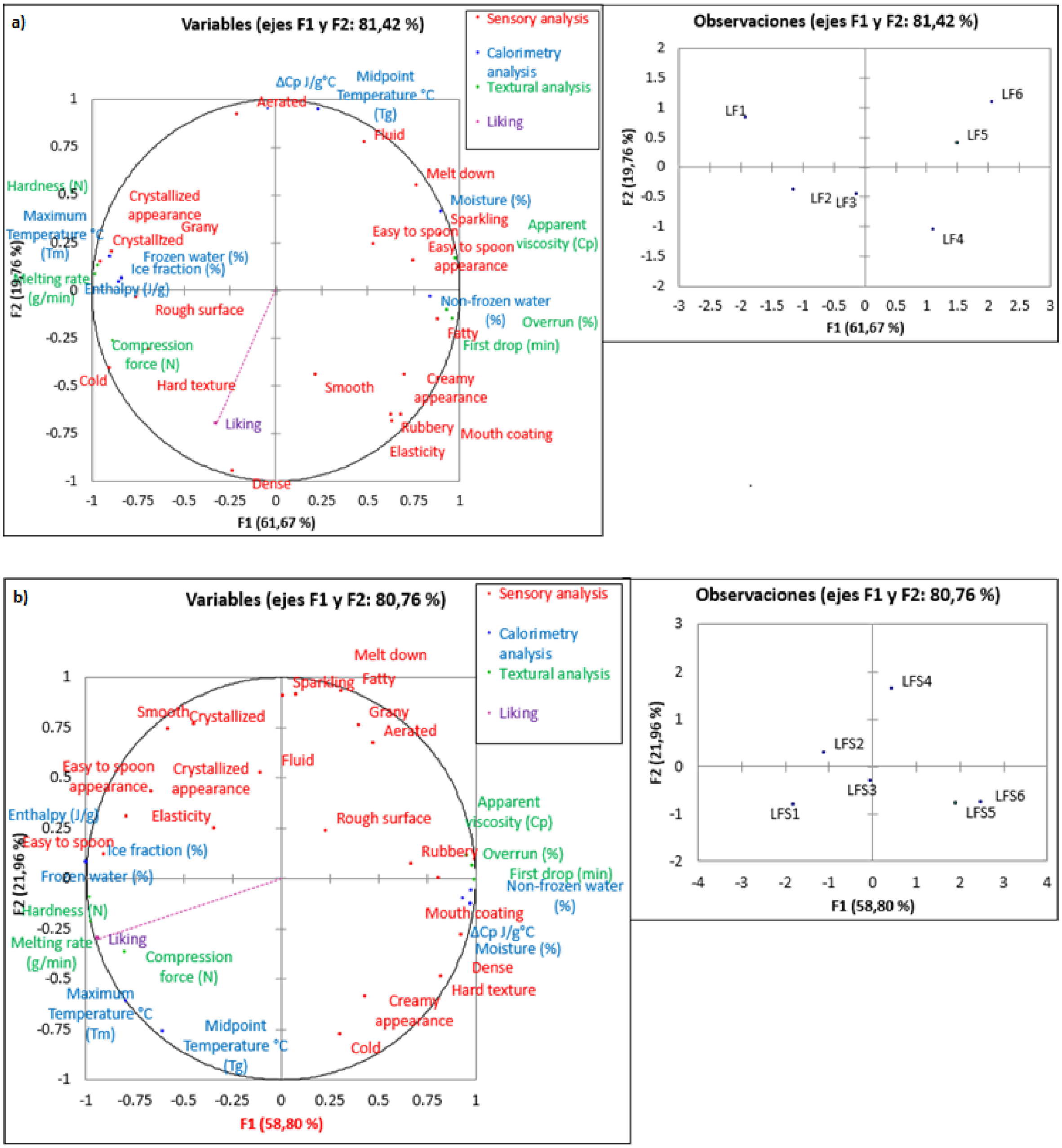

Figure 5. Multiple Factor Analysis of (a) Low-fat ice cream (b) Low-fat and sugar ice cream with agave fructans as a replacer with their respective samples map

4. Conclusions

The quality of ice cream involves many factors associated with sensory, thermal and textural properties, which in turn are related to each other. The use of agave fructans affected the amount of free water and, as a consequence, the quantity and size of ice crystals in ice cream. Based on a triple correlation, sensory attributes, such as texture and appearance crystallized, hard and grainy texture, cold sensation, among others, were related to a formulation that did not contain agave fructans, or contained a concentration of less than 1.2 % of them (LF and LFS 1-3 ice cream formulations). At the same time, these formulations were related to thermal properties such as frozen water, ice fraction, enthalpy and maximum temperature, and textural properties such as hardness, melting rate and compression force. Apparently, the low concentration of agave fructans promoted the increase in ice crystals resulting in an ice cream formulation with hardness and unwanted textures. On the other hand, the samples with concentrations of 1.2 to 3.0% of agave fructans (LF and LFS 4-6 ice cream formulations) showed ice cream formulation with longer melting times, smooth, creamy and fluid texture, and fatty sensation. These properties were directly related to samples with high apparent viscosity and overrun values, as well as low non-frozen water concentrations, high glass transition temperatures and ΔCp values. Finally, the samples that consumers liked most were those that contained higher amounts of fat and sugar, although all the samples had an average score of liking in the hedonic scale. The addition of flavor (such as fruits) and sweeteners could probably increase the liking, but this deserves to be explored in depth in order to optimize the product. Thus, based on these results, it was possible to validate that the use of agave fructans in low fat and low fat and sugar ice cream, enhanced several sensory, thermal and textural properties, making agave fructans a feasible alternative as a fat and sugar replacer.

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7. Artículo 4- Temporal Dominance of Sensations (TDS) and Temporal check all that Apply (T-CATA) for the dynamic characterizing of different ice cream formulations.

Iintroducción.

En este último experimento se llevó a cabo una caracterización sensorial por medio de un análisis descriptivo convencional para evaluar marcas comerciales de helados y helados prototipos reducidos en grasa y azúcar con fructanos de agave. Este análisis se realizó con el fin de comprender la importancia de la formulación del helado, especialmente cuando se reducen los compuestos indispensables como la grasa y el azúcar. Las muestras con grasa de leche o grasa butírica se caracterizaron con los mejores atributos de apariencia, textura, olor y sabor, siendo el helado con características deseables. Los azúcares afectan principalmente el punto de congelación que se reflejó especialmente en texturas indeseables.

Sólo para los helados prototipo se utilizaron métodos sensoriales dinámicos con el objetivo de conocer los diferentes atributos que se perciben en diferentes momentos durante el consumo de helados y cómo se modifican por la presencia de fructanos de agave. Para ambos métodos, TDS y TCATA, vainilla y sabores dulces fueron los atributos más perceptibles durante el período de consumo cuando se usaron 3.0% de fructanos de agave.

Temporal Dominance of Sensations (TDS) and Temporal check all that Apply (T-CATA) for the dynamic characterizing of different ice cream formulations (Dairy Food)

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Abstract

Commercial and prototypes (formulated with agave fructans) ice creams were characterized with a conventional descriptive analysis to understand the importance of formulation and the consequence of fat and sugar reduction that were reflected especially in undesirable textures.

Agave fructans had a better functionality in low fat ice creams when were compared with a normal control. Nevertheless, in low fat and sugar samples, it did not compensate at all, due to attributes as hardness and ice crystals appeared. The use of agave fructans as fat and sugar replacer in ice cream prototypes produced some differences between dominant attributes when we use two dynamic methodologies. For both methods, TDS and TCATA, vanilla and sweet flavors were the most perceptible attributes during the eating period when 3.0% of agave fructans were used reduced fat and sugar ice cream prototypes. In TDS test, the artificial aftertaste appeared later in the consumption, probably for the addition of synthetic or imitation vanilla extracts. Fatty sensation was other dominant attribute in LF prototype

detected during TDS method, probably this sensation was provoked by the increase of creaminess caused by the decrease of free water (produced by the agave fructans and water binding) which is related to the reduction of ice crystals. Ice crystals was an attribute predominate during ice cream consumption especially when fat and sugar were reduced. Possibly, for agave fructans is difficult to compensate both fat solids and the decrease of freezing point caused by sugar. Therefore, It is proposed to increase the agave fructans concentration in ice cream prototypes to avoid the grow ice crystals and enhance texture.

1. Introduction

Ice cream is a complex colloidal food consisting of partially coalescence fat droplets, ice crystals, air cells which are dispersed in aqueous serum phase (Goff, 1997). These structural compounds determine significantly to the perception pattern of texture and flavor. The complete acceptance of ice cream involves a good balance and interaction between compounds that provide a uniform structure which is reflected in a soft, light, and creamy ice cream (Clarke, 2004; Goff, 2002; Soukoulis et al., 2008). Fat and sugar are the main compounds responsible of caloric content same as provide several quality parameters. The reduction or replacement has proven to have a great impact on appearance, textural and sensory perception as well as thermal properties (Bolliger et al., 2000; Clarke, 2004; Goff, 1997; Granger et al., 2005; Hagiwara & Hartel, 1996; McKenna, 2003; Pintor et al., 2018). Currently, the ice cream industry is focused on fat and sugar reduction using compounds that mimic their functionality without affecting the acceptance. Previously, we have studied the use of chicory inulin in low butyric fat ice cream (Pintor and Totosaus,

2013) as well as agave fructans on low fat and sugar ice cream (Pintor et al., 2017). In these works, chicory inulin and agave fructan had a positive effect on viscosity, melting, textural and sensory attributes. Thermal properties were positively affected in the low fat and sugar ice cream using agave fructans as replacers (Pintor et al., 2018).

Oral processing in food is important from two points of view, the ingestion and digestion but it also plays a determinant role in the perception of texture and flavor. These sensory perceptions are dynamic and take place in all stages of the oral processing (Chen, 2015; Foster et al., 2011). During the continuous transformation that involve the first bite to swallowing, many sensory perceptions appear. Cliff and Heymann (1993) suggested that these sensory perception (aroma, taste and texture) are consider dynamic phenomenon that change of intensity throughout the steps of oral processing. The evaluation of ice cream starts from the moment it is observed, nevertheless the definitive evaluation takes place in the mouth, using tongue movements that modified the ice cream structure. There are many investigations about sensory evaluation in reformulated and fat replaced ice cream (Aime et al., 2001; Aykan et al., 2008; Liou and Grun, 2007), protein replacement (Alvarez et al., 2005), sucrose replacement (Akin et al., 2007; Soukoulis et al., 2010), different stabilizers (Guven et al., 2003; Trindade et al., 2007), different fiber flours (Dervisoglu, 2006; Dervisoglu and Yazici, 2006), inulin (El- Negar et al., 2002; Scahller- Povolny and Smith, 1999), or the use of cultured milk (Cruz et al., 2009; Homayouni et al., 2008; Soukoulis et al., 2009). Nevertheless, the impact of sensations over the consumption time has been investigated less despite being very important for the appreciation of ice cream, where the texture changes from a cold solid to a creamy, melting/melted liquid. With the propose of gaining a better understanding about sensory perception some descriptive sensory techniques are designed to provide a measure of sensory perception based on human assessment. Descriptive analysis (DA) is used as standard method that allows the assessors describe an adequate number of sensory attributes associated with a product. The process of DA basically consists of experimental design, panelist selection, term generation and reference standards, evaluation of samples and data analysis (Heymann et al., 2014).

Time Intensity method has been used to show how the dynamics of flavour intensities in ice cream are affected by the fat amount, the type of flavourings (Chung and Grün, 2003; Frøst et al., 2005); or to explore how the temporal profile influences acceptability (Cadena et al., 2011). Additionally, TDS has been applied to study the influence of stabilizers (Varela, et al., 2014).

Currently, several temporal methods have been studied for dynamic sensory characterization of different foods (Cadena et al., 2014). Basically, these methods are focused on the description of sensory characteristics over time. Temporal Dominance of Sensations (TDS) is a technique where the assessor determines from a list of terms the sensation that captures their attention at each moment of the evaluation. Along the evaluation, each time the dominant attribute changes, the panelists have to select the new dominant sensation and score it. (Pineau et al., 2009). The concept of “dominance” has change throughout the time. Nevertheless, Varela et al. (2017) have conceptualized this term based in trained assessors and consumers and they found that dominance is a complex construct related to multiple aspect of perception that could modified the interpretations of the results. The results obtained were represented in TDS curves which consist of the dominant rate of attributes (Y-axis) against time (X-axis) (Cadena et al., 2014).

TDS method has been used to study the dynamic perception of different products, such as wine (Meillon et al. 2009; Sokolowsky and Fischer 2012), blackcurrant squashes (Ng et al. 2012), coffee (Dinnella et al. 2013), water (Teillet et al. 2010), gels (Labbe et al. 2009), dairy products (Bruzzone et al. 2013; Pineau et al. 2009), wheat flakes (Lenfant et al. 2009), biscuits (Laguna et al. 2013), fish sticks (Albert et al. 2012), extra-virgin olive oil (Dinnella et al. 2012), and salmon sauce (Paulsen et al. 2013). In a previous investigation, different ice cream formulations (contained milk, cream, egg, and hydrocolloids) were evaluated by the Temporal Dominance of Sensations (TDS) and iciness, coldness, creaminess, roughness, gumminess, and mouth coating (Varela et al., 2014).

Recently, a multi-attribute temporal method known as Temporal-Check-All-That-Apply (TCATA), which is an extension of CATA questions, was introduced by Castura et al. (2016). It has proven to be easy to perform and no tedious (Ares et al., 2015). In this technique, assessors are requested to select from an attribute list, all those that apply to describe sensations perceived at each moment of the evaluation as well as the possibility to uncheck attributes when they are no longer applicable moment to moment. Many products have been studied with TCATA methodology, such as orange juice, strawberry yogurt, French bread, chocolate-flavoured milk, chesse, salami and mussels and cosmetics emulsions (Ares et al., 2015; Boinbaser et al., 2015; Castura et al., 2016; Olivera et al., 2015). This differs to the TDS methodology that only allow assessors to select attributes that catch their attention along time. This difference enables TCATA to provide more detailed description of dynamic sensory profile during consumption than TDS. Other disadvantage of TDS method is that assessors may lose attention when they try to decide which modality is more relevant (for example, either form texture or flavour) and when the attribute dominance is not clear (Varela et al., 2017). A possible solution for this inconvenient is running TDS in separate steps, such as Agudelo et al. (2015) have proposed. TDS and TCATA methodologies, and their comparison, have already been studied; however, there is not enough information yet about the application of these techniques in complex product such as ice cream. Therefore, the principal aim of this work is to better understand static and dynamic sensory profiles of ice creams reduced in fat and/or sugar, and those formulated with agave fructans as replacers.

2. Material and methods

2.1. Commercial and prototype ice cream samples

For this study the samples were obtained from different ice cream shops from Mexico City (commercial ice cream), and other samples were prepared in the sensory evaluation laboratory of Universidad Autónoma Metropolitana (ice cream prototypes). The sensory evaluation of the ice cream was carried out in the sensorial evaluation laboratory of the Faculty of Chemistry of the Universidad Nacional Autónoma de México. For the selection of attributes, the samples with the most different characteristics were used: three commercial samples and two prototypes.

The dynamic methods TDS and T-CATA were applied only for the ice cream prototype samples. The commercial vanilla ice creams were: *Nestle*, *Holanda*, *Precissimo*, *Valley Foods*, *Häagen-Dazs*, *La Michoacana* and *Santa Clara* (they are identifying as whole composition) contain butyric or vegetable fat, sugar, milk and whey protein, emulsifiers and hydrocolloids), *Deslice cream* (gluten and lactose free) and *Vital´ice* (without sugar and gluten, added with probiotics) (Table 1)

The ice cream prototypes were: *normal and reduced control (control LFS)*, prototypes *LF* (low fat with agave fructans) and *LFS* (low fat and sugar with agave fructans), based on previous studies (Pintor et al., 2013; Pintor et al., 2017) (Table 2).

Table 1. *Composition of ice cream samples*

Type of ice cream	Commercial brand	Composition	Shop place
Whole composition	Nestlé	Milk solids, skim milk, sugar, glucose syrup, vegetal fat (6.9%), gums, emulsifiers, flavorings and colorants.	Comercial Mexicana
	Holanda	Water, skim milk, sugar, vegetal fat (6.9%), milk solids, emulsifiers, flavorings and colorants.	Comercial Mexicana
	Preccisimo	Water, sugar, vegetal fat (6.8%), solids milk, gums, emulsifiers, flavorings and colorants.	Comercial Mexicana
	Valley Foods	Whole fluid milk, milk cream, sugar, water, milk solids, corn syrup, emulsifiers, thickeners, flavoring and colorants.	Comercial Mexicana
	Häagen-Dazs,	Milk cream, skim milk, sugar, yolk, flavoring and milk fat (17.0%).	Comercial Mexicana
	La Michoacana	Without label	Polanco mall
	Santa Clara	Without label	Coyoacán centro

	Normal control	Water, sugar, butyric fat, milk protein, whey protein, vegetal fat, stabilizers, and emulsifiers.	Prototype UNAM
Gluten and lactose-free	Deslice cream	Water, skim milk (1.1%), lactase, sugar, vegetal fat (11.4%), solids milk, emulsifiers, stabilizers, flavoring, colorants and conservatives.	Chedraui
Without sugar and gluten, with probiotics	Vital´ice	Water, whole milk, agave syrup, cream powder, sweeteners, stabilizers, flavoring and encapsulated lactobacillus.	City Market
Fat reduce	Prototype LF	Water, sugar, butyric fat, milk protein, whey protein, vegetal fat, agave fructans, stabilizers, and emulsifiers.	Prototype UNAM
Fat and sugar reduce	Prototype LFS	Water, sugar, butyric fat, milk protein, whey protein, vegetal fat, agave ructans, stabilizers, and emulsifiers.	Prototype UNAM
	Control LFS	Water, sugar, butyric fat, milk protein, whey protein, vegetal fat, agave fructans, stabilizers, and emulsifiers.	Prototype UNAM

***Table 2**. Prototype ice cream composition*

Compounds	Normal control	Reduce control LFS	Prototype LF	Prototype LFS
(%)				
Milk protein	8	8	8	8
Whey protein	4	4	4	4
Butyric fat	**10**	**7**	**7**	**7**
Vegetable fat	4	3.5	3.5	3.5
Sugar	**15**	**13.2**	**15**	**13.2**
Establizer	0.5	0.5	0.5	0.5
Emulsifier	0.25	0.25	0.25	0.25
Agave fructans	**0**	**0**	**3**	**3**
Water	58.25	63.55	58.75	60.55

2.1.1. Ice cream prototypes manufacture

For preparing the ice cream prototypes (Table 2), all the dry ingredients, sugar, non-fat dry milk and whey protein concentrates (DILAC S.A de C.V) and emulsifiers (sorbitan and glyceryl monosterates, ARCY S.A de C.V. México) were hydrated in water at 60°C to disperse anhydrous milk fat (referred as butyric fat) and vegetable fat (La Mixteca, México). Agave fructans (Vaserco, S. de R.L de C.V., Guadalajara, Mexico) were employed to replace both fat and sugar. The homogenized mix was pasteurized at 70°C for 30 minutes and stored at 4°C during 24h. The ice cream mix (non-frozen mixture) was frozen in a 2 quarters frozen-ice cream CIM 50RSA machine (Cuisinart, East Windsor) for 20 minutes. Vanilla flavor was added during the mix.

2.2. Selection and trained panel

Selection of assessors was based on aspects as health and eating habits, threshold of odors and basic tastes, identification and recognition of odor, test of memory, as well as discriminative flavor test. Twenty-two assessors were recruited and trained to ensure the effectiveness on discrimination, quantitation, repeatability and agreement of results.

2.3. Conventional Descriptive Analysis

The attribute list was generated in two sessions, agreeing on a list covering aspect, odor, texture, flavor and residual sensation. The results were quantified on base the number of times mentioned, specified to describe the samples, synonyms homogenization and concordance. After this, the descriptors and definitions were agreed upon by the assessors. In other session that two commercial and two ice cream prototypes were selected, the assessors were asked to use a structured scale of 7-points labelled 0= is not present, 7= very intense to evaluate the attributes. The results were used to identify the attributes that showed a better dispersion in the punctuation of assessors and to define which attribute required the use of standards to anchor. The final list (Table 3) contained of five aspect attributes (color, bright, crystals, porous, creamy), two odor attributes (vanilla and caramel), ten texture attributes (hard, viscous, creamy, ice crystals, sandy, gummy, melt down, mouth coating, sparkling and dense), five flavor attributes (vanilla, sweet, milk, caramel and salty) and one artificial residual sensation.

.**Table 3**. Attributes generated by the trained panel and their definitions

.2.4. Temporal Dominance of Sensations (*TDS*)

	Attribute	*Abbreviation*	*Definition*
Appearance	Color	Color	Sensation produced by the stimulation of the retina created by the light rays of several wavelengths
	Brightness	Brightness	Describes the amount of light reflected by the sample
	Presence of crystals	Presence_cr	Visible ice particles proper to the freezing of the sample
	Porosity	Porosity	Geometrics attributes in which small holes are observed in the sample
	Creamy	Creamy	The sample is visually perceived as smooth texture without defects and it melts uniformly
Odor	Vanilla	Vanilla_o	Characteristic vanilla odor. Sweet standard
	Caramel	Caramel_o	Note to toffe, lightly toasted hard candy
Texture	Hardness	Hardness	Force required to penetrate the sample with the incisors
	Viscosity	Viscosity	Attribute related to resistance to flow. Corresponds to the force required to suck a liquid from a spoon on the tongue or to spread it on a surface
	Creamy	Creamy	It is the velvety sensation that the sample provokes in the mouth
	Ice crystals	Ice crystals	Presence of frozen water particles that are perceived in the mouth
	Sandy	Sandy	Sensation related to the presence of small particles
	Gummy	Gummy	Perception of gummy, chewable texture
	Speed to melt	Speed_ m	Speed of sample when it melt in the mouth with respect to time
	Fatty sensation	Fatty_s	Amount of fat that is perceived as a layer around the mouth
	Frothy	Frothy	Presence of air burbles
	Dense	Dense	Sensation that is perceived by the influence of solids in the sample
Flavor	Vanilla	Vanilla_f	Characteristic vanilla flavor
	Sweet	Sweet_f	Characteristic basic taste of aqueous solution with sucrose 0.46 (g/100g)
	Milk	Milk_f	Characteristic test of slightly sweetened milk
	Caramel	Caramel_f	Hard caramel flavor, slightly toasted and alcoholic
	Salty	Salty_f	Characteristic basic taste of salt
Aftertaste	Artificial	Artificial	Flavor to vanilla artificial flavoring. A plastic note can also be presented

The principal aim for TDS task was to detect the dominant attribute in a certain time during evaluation, followed by quantitation. The evaluation was conducted following the TDS approach presented by Pineau et al. (2009). Trained sensory panelist (n=22) were used for the analysis. In preliminary sessions, descriptive analysis enabled to select those attributes that presented significant differences for all the samples emphasizing the ice cream prototypes. Ten attributes (texture, flavor and aftertaste) whit their definitions were used to the formal assessment (Table 4). The intake time was 8 minutes that started from the moment the panelist puts the spoon with the sample in his/her mouth and ended until no sensation was perceived. Samples were assessed in triplicated. In the formal assessment, the questionnaire was divided in two parts; in the first one only flavor and aftertaste descriptors were shown and in the second one texture attributes. The procedure presented on the computer screen was as follow: assessors were asked to put a spoon with enough sample in their mouth and press “START”; subsequently, they selected the dominant sensation and gave a value in a structured scale in each moment of evaluation. At all times, only one attribute was allowed to be selected (the dominant one). When assessors felt no sensation they had to click the “STOP” button. Between each sub-group assessors were asked to rinse their mouth with water and eat a salty cracker.

***Table 4**. Sensory attributes for prototypes ice cream in TDS task*

	Term	Definition
Texture	Hardness	Force required to penetrate the sample with the incisors
	Ice crystals	Presence of frozen water particles that are perceived in the mouth
	Sandy	Sensation related to the presence of small particles
	Speed to melt	Speed of sample when it melt in the mouth with respect to time
	Fatty sensation	Amount of fat that is perceived as a layer around the mouth
	Frothy	Presence of air burbles
	Dense	Sensation that is perceived by the influence of solids in the sample
Flavor	Vanilla	Characteristic vanilla flavor
	Sweet	Characteristic basic taste of aqueous solution with sucrose 0.46 (g/100g)
Aftertaste	Artificial	Flavor to vanilla artificial flavoring. A plastic note can also be presented

2.5. Temporal check all that apply (TCATA)

The methodology was described by Castura et al. (2016). In a preliminary test, assessors confirmed the descriptors that were selected from the Descriptive Profiles, and then the definitive questionnaire was designed. The TCATA list included the same ten attributes as in the TDS task. The principal objective was that assessors were checking all the attributes that describe the sensory characteristics of the prototypes ice cream samples at each moment of the evaluation (each 20 seconds during 5 minutes) and to uncheck the terms when they no longer applicable. All attributes could be checked more than once, and they could be repeated several times during the evaluation.

2.6. Data analysis

The software FIZZ Acquisition, 2.51 was used for questionnaires design and to perform each test, while the results analyses were performed using FIZZ Calculations, 2.50 (Biosystems, 2007, Couternon, France). For conventional descriptive methodology, it was made an analysis of variance with a level of significance of 95% (two- way ANOVA) to determine if exist significant differences between the evaluated attributes or between the assessor's dispersions. To complete the analysis, the standard deviation and the coefficient of variation for all the attributes were calculated to assure they were lower than 40%, indicating a satisfactory result. In addition, a Principal Component Analysis (PCA) was used to measure the interactions of the samples between the evaluated attributes.

For TDS analysis data, it was collected for each panelist run all the attributes chosen as dominant and the times when the dominance started and stopped. Time standardization was applied to remove assessor noise (Lenfant et al., 2009). For each point of time the proportion of run which were identified as dominant were plotted against time. The TDS curves contained two additional lines which showed a significant and proportion level as statistical criteria to know the attributes that were pointed as significant.

TCATA curves were plotted as the citation proportion of each attributes at a given time.

3. Results explanation.

Conventional descriptive analysis results are described below; however, before carrying out the definitive evaluation, an analysis of variance using attributes and assessors as factors was performed in order to validate a better reliability in the results. Samples were mapped in relation to their descriptors by PCA, that were performed separately depending on their composition (reduced and commercial samples). The attributes that showed significant differences in the sample comparison of the three groups (whole, reduced and normal samples) were selected for TDS and TCATA methodology (Tables 3 and 4), whose results are described afterwards.

3.1. Conventional descriptive analysis

3.1.1. Prototype ice cream samples

The figure 1a shows the PCA for appearance attributes results of reduce samples ice cream. 67.5% of the total variance was explained by the two principal components. The prototype LFS was correlated positively to the components 1 and 2, it was characterized by the presence of crystals in addition of brightness and porosity. While the sample Deslice was correlated positively with the component 2 and was related to creamy attribute. Finally, the experimental controls (normal and LFS) were correlated positively to the component 1 and were characterized principally by color attribute. In the PCA for texture (Figure 1b), only the 54.3% of the total of variance was explicated by the components and solely the prototype LFS was correlated positively with ice crystals and hardness. In the figure 1c was plotted the PCA for flavor, odor and aftertaste in which only the 48.2% of total variance was explained by the components. The commercial samples Deslice, Vital´ice and the normal control were correlated positively with the components 1 and 2, same as they were described as vanilla and caramel odor, and caramel flavor attributes. The prototypes LF and LFS were characterized in low intensity as salty flavor. In the annexes was observed that LF is very similar to normal control in terms of appearance. The use of agave fructans reduced the amount of ice crystals and increase milk flavor. By other hand, in attributes related to appearance, the prototype LFS compared with control LFS resulted in an increase of ice crystals. Texture attributes as dense, gummy, viscosity ice crystals and harness, increase despite of the presence of agave fructans. Nevertheless, milk favor increase.

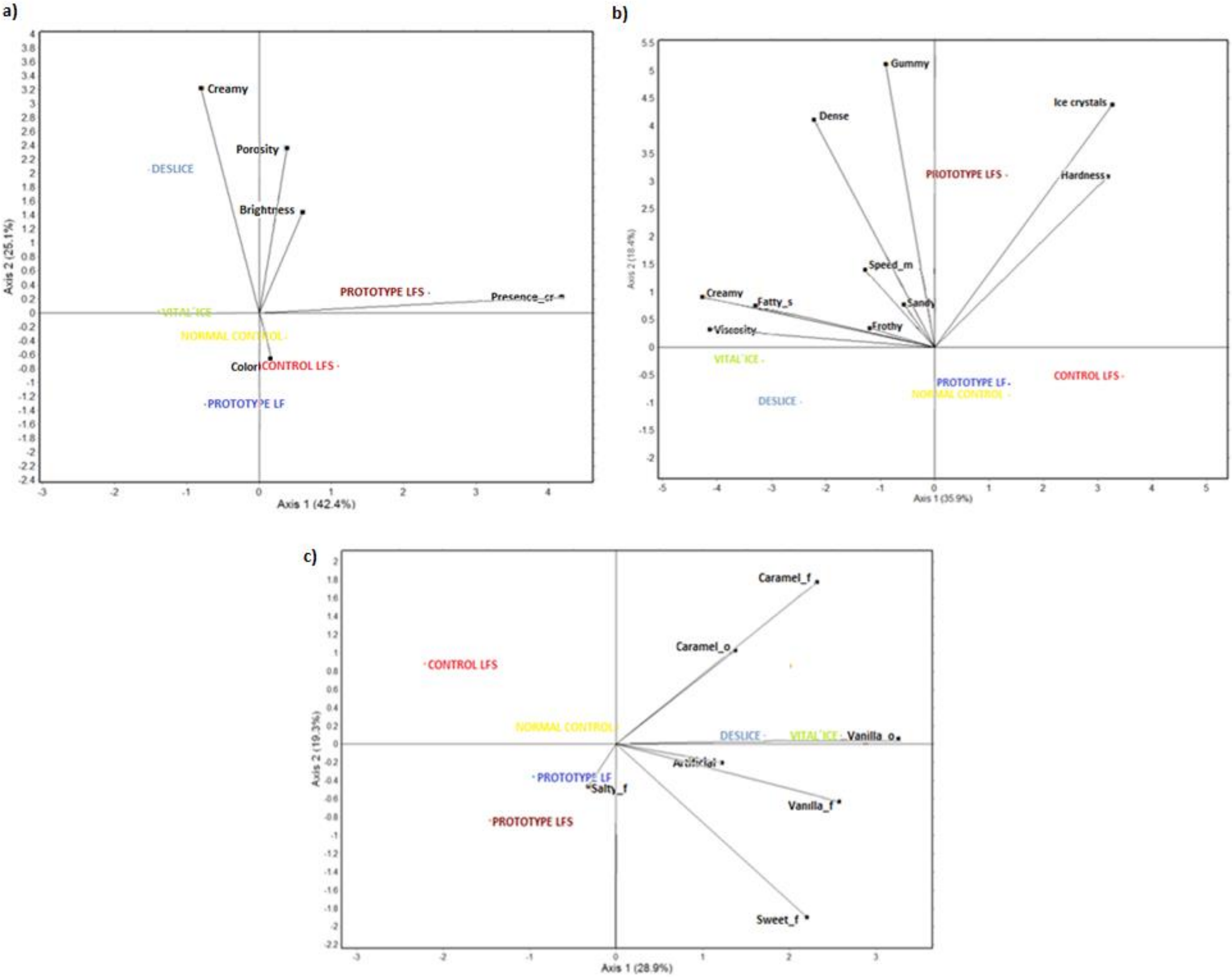

Fig.1. Principal Component Analysis (PCA´s) for a) appearance, b) texture and c) flavor, odor and aftertaste attributes of the reduced samples (commercial and ice cream prototypes).

3.1.2. Commercial ice cream samples

The Figure 2 a shows commercial ice cream samples with whole composition (Table 1)) and their correlation with some appearance attributes. The 65.9% of total variance of the results from the PCA was explicated. The commercial sample Michoacana was correlated positively with both components and the assessors decided related it to color and brightness. The samples Valley food and Holanda were correlated positively with the component 1 and negatively with the component 2, they were characterized by porosity and presence of crystals. Finally, Nestlé was correlated positively with the component 2 and it was related to creamy. The PCA that correspond to texture attributes, have a high explicability of total variance compared to the other analysis (83.3%). In the results it was observed that all the sample were correlated with at least attribute; for example, Valley food was correlated positively with sandy and ice crystals. Precissimo and Holanda were characterized only by speed to melt. La Michoacana ice cream was correlated positively with the component 2 and it was the sample with more attributes mentioned being these: viscosity, hardness, fatty sensation, gummy, dense and creamy. The saples Santa Clara, Häagen-Dazs and Nestlé were correlated negatively for both components and they were characterized by frothy (Figure 2 b). To end with PCA analysis, the attributes for flavor, odor and aftertaste results were plotted (Figure 2 c). 81.3% of the total variance of the samples were explicated by both components. The sample Häagen-Dazs was correlated positively with the component 1 and it was characterized by vanilla flavor. Precissimo and Nestlé were related to vanilla flavor and odor. Finally, La Michoacana and Valley Food were correlated negatively for both components, and they were marked as caramel, sweet and salty flavor, caramel odor and artificial aftertaste. Base on analysis of results, it will try to make a global analysis for appearance, textural, flavor, odor and aftertaste attributes that characterized the samples for both group (prototypes and commercial ice cream).

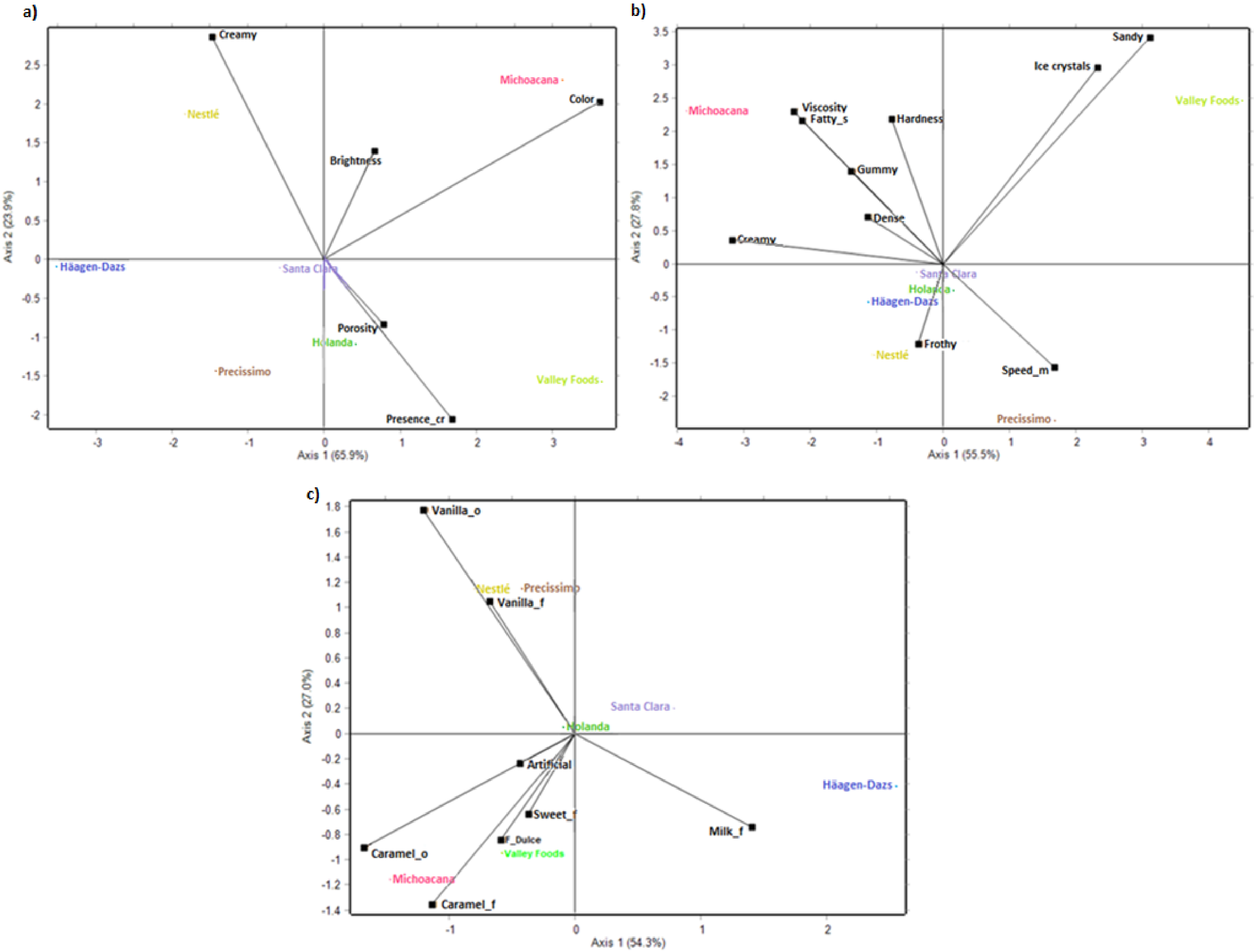

Fig.2. Principal Component Analysis (PCA´s) for a) appearance, b) texture and c) flavor, odor and aftertaste attributes of whole composition samples (commercial samples).

3.2. Dynamic Sensory Profiling analysis (TDS and TCATA)

3.2.1. Temporal Dominance of Sensations (TDS)

The figure 3 (a-d) shows the TDS graphs for the ice cream prototypes. Each curve represents the attributes chosen as dominant at each moment during the evaluation (Varela et al., 2013). As mentioned before, the selected attributes were those that showed significant differences between ice cream samples after a two- way ANOVA after the descriptive analysis. The curve for normal control, reduce control and the prototype LF (Figures 3 a-c) showed that flavor attributes were the first dominant perception to appear, specifically vanilla flavor was dominant at the beginning of the oral processing, followed by sweet flavor. These two attributes lasted at least half of the evaluation. For normal control (Figure 3 a), artificial aftertaste was significant, and this attribute lasted until the end of the evaluation. Fatty sensation was dominant for the prototype LF (Figure 3 c). Nevertheless, the dominance rate for these attributes were higher than the significance level, but their values were medium, which mean that the attributes did not found very high consensus in the evaluation. In the prototype LFS (Figure 3 d), vanilla flavor was the first attribute dominant, which, lasted throughout 60% of the TDS evaluation and showed a high dominance rate compared to the other samples. The term hardness was hardly significant at the beginning of the oral process. The texture attribute ice crystals were dominant and enduring for all the evaluation time.

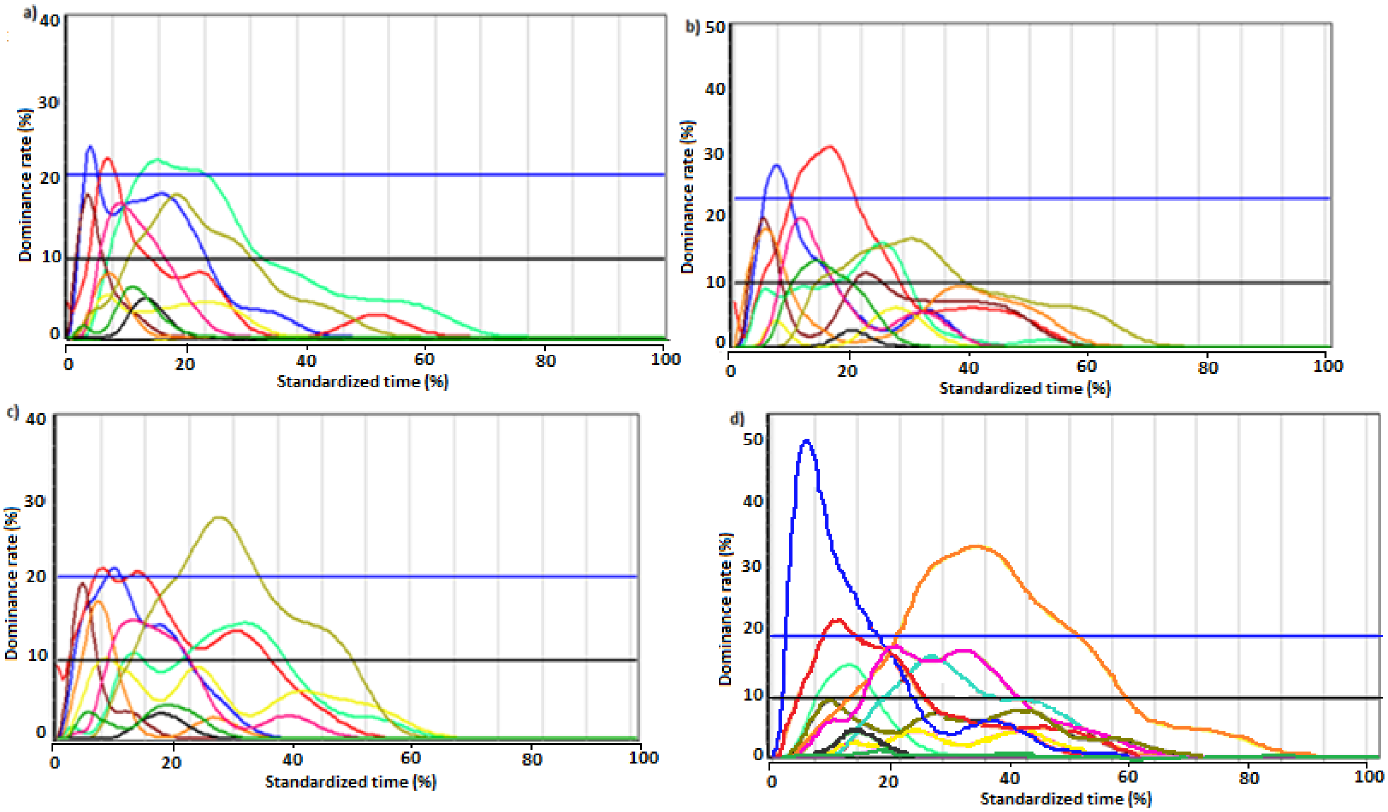

Fig. 1. Temporal Dominance of Sensations (TDS) curves for the prototypes ice cream samples: a) Normal control, b) Reduce control, c) Prototype LF and d) Prototype LFS ▬Vanilla flavor,▬Sweet flavor,▬Artificial aftertaste,▬Hardness,▬Ice crystals,▬Sandy,▬Speed to melt,▬Fatty sensation, ▬Frothy, ▬Dense,▬ Chance level,▬ Level of significance (5%).

3.3. Temporal Check All That Apply (TCATA)

For each sample line plot were created by calculating the proportion of judgment (i.e. assessor x replicated) for which it was selected for describing each sample at each time of the evaluation (Vidal et al., 2017). Temporal profile for all the samples (Figure 4 a-d) were characterized by sweet flavor but in different duration in the time of eating period. For normal control (Figure 4 a) sweet flavor was applicable for approximately 28 seconds, while vanilla flavor and fatty sensation were significant but with a short duration (16 and 21 seconds respectively). For reduce control and the prototype LF samples (Figures 4 b and c), only sweet flavor was significant for both, but for the first one showed a low time of eating period (42 seconds approximately) compared with the second one sample which had 83 seconds approximately of duration. For prototype LFS sample (Figure 4 d), sweet and vanilla flavor had a duration of 69 and 48 seconds, respectively. For these last three samples the citation proportion ranges from 30 to 40.

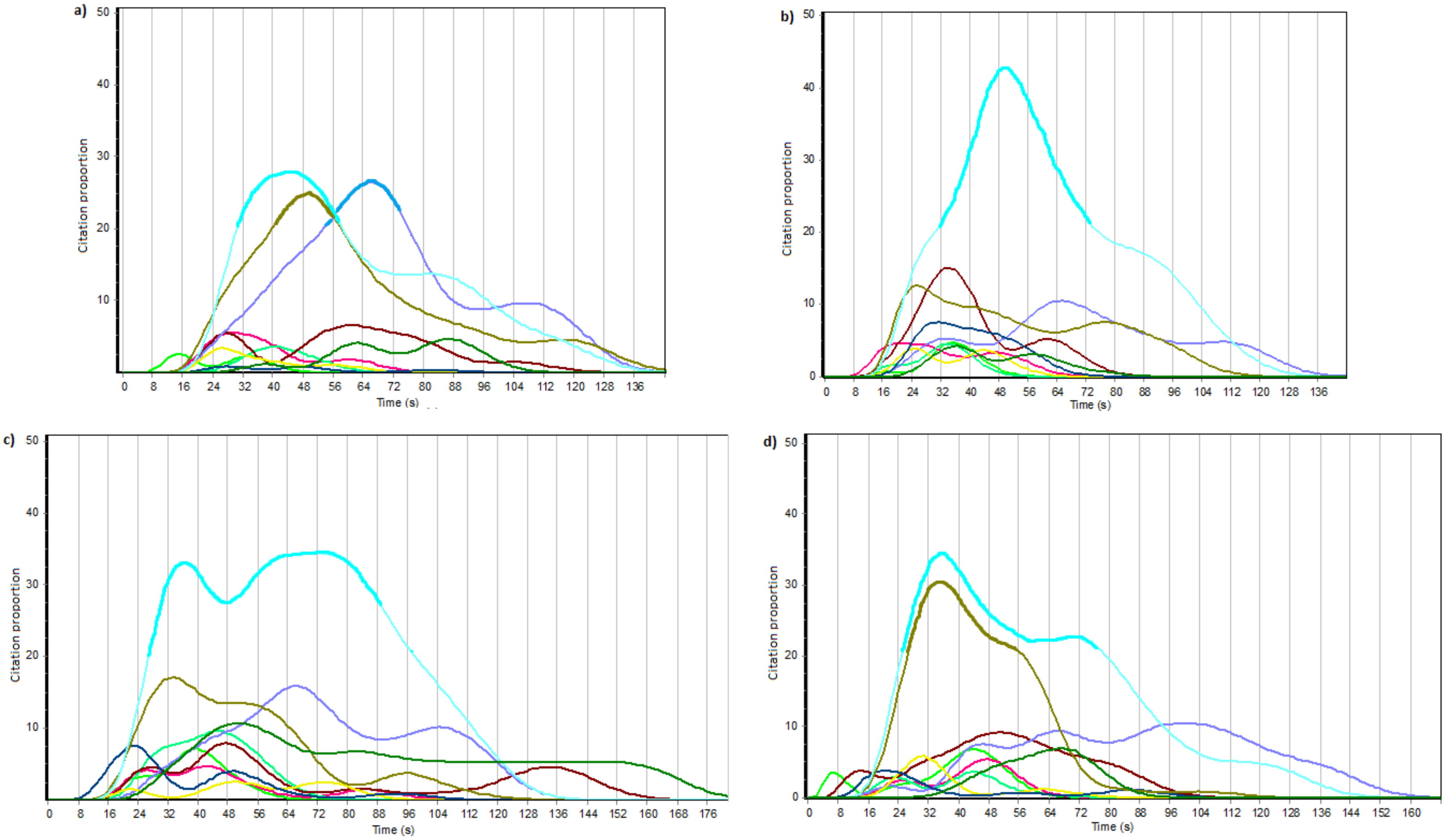

Fig.5. Temporal check-all-that apply (TCATA) curves for prototype ice cream a) normal control, b) reduced control , c) Prototype LF and d) prototype LFS: —Hardness, —Ice crystals, —Sandy, —Speed to melt, —Fatty sensation, — Frothy, — Dense, — Vanilla flavor, — Sweet flavor, —Artificial aftertaste.

4. Results discussion

4.1. Conventional descriptive discussion

4.1.1. Appearance

It is maybe the characteristic more important for the assessor at the time of choosing any food. It must be attractive at sight, with special attention on packing, optical properties and physical shape of food (Alvarez, 2009). Comprising color, shape, size, gloss, uses the optical sense (Bourne, 2002). Among the attributes selected for appearance, artificial colorant was choosing for the prototypes normal control, control LFS (Figure 1 a) and the commercial sample La Michoacana (Figure 2 a). The color present in sample is probably due to the addition of colorant during the ice cream manufacture. The color of vanilla should be attractive, uniform, pleasing and typically of the specific flavor (French, old fashioned, vanilla bean, etc.). As long as the shade of color reasonably resembles the natural color (b-carotene pigment) of cream and is neither too pale nor too vivid, color criticisms are generally resisted for vanilla-flavored products (Alvarez, 2009). In previous studies, the color and brightness of low fat ice cream not were modified by the use of inulin as fat replacer (Pintor et al., 2013). The samples identified as porous and with presence of crystals were the prototype LFS (Figure 1 a), Valley Food and Holanda (Figure 2 a). These two last commercial samples have a similar composition, the problem is that the label does not specify the quantity of the components. The porosity is an attribute that was perceived immediately as small holes in the sample and probably was directly related to the quantity of air incorporated during the shake and freeze of mix. This attribute is may associate the presence of foam with excessive overrun, even though this defect may not be associated with high overrun, but more often (or rather) with some of constituents used in the mix. The presence of crystals is due to the increase of size and quantity of ice crystals, principally by the high amount of free-water. Deslice and Nestlé (Figure 2 a) were characterized as creamy. The creaminess involves many factors principally associated with textures, but others have to do with flavor (mainly vainilla, sweetness and fat related flavor) (Antmann et al., 2011). In the case of ice cream, creaminess is primarily associated with a high fat and protein content and is usually described as the perception of a somewhat soft mass that melts gently without detectable particles appearing in the mouth. These parts will be better explained when talking about the frothy, crystallized and creamy textures.

4.1.2. Texture

In sensory analysis the texture is other important parameter which evaluate the response of tactile to physical stimuli that result from the contact between some part of the body and the food. In the texture evaluation infers tactile, sight and sound sense (Bourne, 2002). Sandy was an attribute found in Valley Food ice cream (Figure 2 b). Sandy texture is one of the most know defects and easy to detect in ice cream. This undesirable texture is distinguishing when the sample melts, there remains in the mouth fine, hard, uniform particles that suggest fine sand, and are crystals of lactose. Lactose has a relatively low solubility; as a result, it can crystallize as monohydrate (i.e. for each lactose molecule there is also a water molecule in crystals). Exist many was to ensure that is about lactose crystals instead of ice crystals, the principal is that it not melt easily in the mouth during the consumption. Other ways are pressing a thin layer of ice cream against the roof or the mouth, bringing the teeth together with some portion or pressing small quantity between the thumb and forefinger. To avoid this defect in Valley food sample could calculating the expected lactose concentration in the matrix of the formulation, since about 50% of lactose is found in the skimmed milk powder (Alvarez, 2009; Clarke, 2004). Lactose crystallization has been studied from two point of view, the first one due to the unfrozen phase become supersaturated and the second by effect of temperature fluctuation (Livney et al., 1995).

The presence of crystals (appearance attribute), ice crystals and hardness (textural attributes) were present for the prototype LFS (Figures 1 a, b), which it was reduced in fat and sugar. At the same way Valley food was characterized as ice crystal texture and La Michocana as hardness (Figure 2 b). Ice crystals is the most commonly encountered texture defect in ice cream. Ice texture is due to comparatively large particles of frozen water; each ice crystal is sufficiently large that the coarseness is obvious. The assessors can considerate them with an ice crystals structure, a feeling of unusual coldness within the mouth, a simultaneous lack of a smooth, velvety character, and a frequently associated rough and crystallized visual effect. The way to detect the presence of ice crystals is placed the sample between the upper and lower incisors a temporary resistance is exhibited before the incisors are finally permitted to come together (Alvarez, 2009). The force exerted to break either ice crystals, burbles and emulsified fat globules in ice cream is called hardness and it is determining by the quantity and size of ice crystals, formed during the freezing and storage (Akesowan, 2008; Goff, 1997). The ice crystals contribute to hardness ice cream, but the biggest ice crystals increase considerably this attribute. A lot of disperse solids provoke more resistance to apply a force and consequently hard ice cream textures (Muse and Hartel, 2004). In this sense, the fat substitutes (as water binding water) decreased the hardness in ice cream (Roland et al., 1999; Karaca et al., 2009). The possible causes of an ice texture could be the faulty formulation, inadequate protection against heat shock, ineffective stabilization and/or emulsification, inadequate hydration of dry components, incomplete protein hydration and homogenization, temperature fluctuation, extended interval between freezing, packaging, and/or transfer to the hardening system, extended storage and distribution times, and the reduction of compounds as is the case of this work.

In ice cream, fat is responsible of emulsion formation, increasing viscosity in the serum phase, creating a film on the surface of the air cells that promote stability in the ice cream, decreasing the melt time, reducing the growth and size of ice crystals, providing texture, palatability, creaminess, releasing flavor molecules and enhancing mechanical properties (Adapa et al., 2000; Akalin et al., 2008; Bolliger et al., 2000; Chung & Grun, 2003; Goff, 1997; Goff, 2002; Goff & Hartel , 2013; Granger et al., 2005; Roland et al., 1999; Sung & Goff, 2010). On the other hand, sugars in ice cream have two principal functions: to provide sweetness and control the amount and size of ice crystals, affecting ice cream softness. Sugar has the ability to decrease the freezing point of the serum phase and therefore reduce the amount of ice caused by crystallization and recrystallization. (Goff & Flores; 1999; Hagiwara &

Hartel, 1996; McKenna, 2003). The decrease of fat or sugar provoke principally undesirable ice cream textures and appearance. Nevertheless, to counteract negatives effect to the reduction of sugar and fat, it has been used compounds that mimic their functionality. For example, inulin was employed in ice cream for its binding water properties to form a particulate gel network, it acts like a cryoprotectant-ice crystal size reduction during freezing and storage, in addition to improving other textural and sensory properties (Akin et al., 2007; Aykan et al., 2008; El-Nagar et al., 2002; Karaca et al., 2009; Lobato et al., 2009; Schaller-Povolny and Smith, 1999; Soukoulis et al., 2009). The use of agave fructans has not been widely studied in ice cream, nevertheless, some works have been carried out about agave fructas as fat and sugar replacer, where concentration of 3.0% (approximately) improved thermal properties in low fat and low fat and sugar ice cream. (Pintor et al., 2018). Probably the increase of agave fructans concentration (above 3.0 %) in prototype LFS improves properties related to texture, since agave fructans compensate on one hand the butyric fat reduction (added water is retained by branched agave fructans structure), and on the other hand, sugar reduction (agave fructans as hygroscopic material that reduce freezable water), provoking softer textures (Espinosa et al., 2012).

The commercial samples Holanda and Precissimo (Figure 2 b) were characterized with speed to melt attribute. These sample have in common the vegetable fat content (6.9 and 6.8% respectively). In USA, ice cream must contain at least 10% milk fat and 20% total milk solids and must weigh a minimum of 0.54 kg L. Time ago the ice cream with vegetable fat it was not consider “ice cream”. Currently exist many ways to characterize ice cream according fat and solids content. Normally, premium ice cream is made from the best quality ingredients and has a relatively high amount of dairy fat and low amount of air. On the other hand, economic ice cream is made from cheaper ingredients (e.g. vegetable fat) and contain more air (Clarke, 2004). Melting properties are influenced by the three structural components that make up the dispersion phase of ice-cream: ice, air and fat. The melting properties are expensed of gradual heat penetration from environment (in this case mouth) to ice cream interior provoking ice crystals fusion making that the water contained in ice cream flowed through out the foamy structure and finally drop. (Muse and Hartel, 2004). A stable ice cream resists or delay structural changes in a dynamic environment (Goff, 1997). Melting rate was normally inversely proportional to overrun (air content), with lower melting rate producing higher overrun values and vice versa (Sakurai et al., 1996; El-Nagar et al., 2002; Muse and Hartel, 2004; Sofjan and Hartel, 2004; Akalin, A. & Erişir, 2008). In turn, higher overrun values result in slower melting, since air cells act as an insulator medium (Sakurai et al., 1996; Caillet et al., 2003; Marshall et al., 2003; Akalin and Erişir, 2008). A good distribution and minimization of air bubble size through the ice cream matrix contributes its stability (Goff, 2002). In other study, fat level and not the fat type affected the melting behavior of ice cream samples (Hyvonen et al., 2003), where a higher fat content seemed to retard the melting rate of the ice cream (Karaca et al., 2009). Other parameters such as globule interactions and/or fat crystallization may also influence the melting behavior of ice cream mixes (Granger et al., 2005). In the drip losses fat content was directly correlated with the maximum meltdown rate (Koxholt et al., 2011). Other compound that influence the speed to melt of ice cream are gums or hydrocolloids. This ingredient has the ability to retain larges amount of free water, hence melting rates (Regand and Goff, 2003; Akesowan, 2008). For example, the interaction between iota carrageenan, locust bean gum and carboxymethylcellulose modified positively the ice crystals formation and improved melting characteristics (Pintor and Totosaus, 2012).

The sample La Michoacana is a sample without specifications in the label. It was described with texture attributes as viscous, gummy, fatty sensation, dense and creamy (Figure 2 b). Viscosity is defined as the internal friction of a fluid or its tendency to resist flow (Bourne, 2002). Viscosity is a parameter that is detected in the ice cream mix (prior to freezing) or when the ice cream is melt in the mouth. The increase of viscosity can be influence by different factors; the use of gums or hydrocolloids which interact with liquid components of ice cream mix. This effect is caused by two reasons, the contribution of solids to the aqueous phase and the water-binding effect of hydrocolloids which form a gel-like network that modify the viscosity and enhance the emulsion stability (El-Nagar et al., 2002; Akin et al., 2007; Soukoulis et al., 2009). For example, carrageenans increased the mix viscosity in ice cream due to the capacity to extend their configuration interacting with other polysaccharides, provoking a polymer volume increase that result in more water junction zone, due to negatives charges sulphate group interactions (Pintor and Totosaus, 2012). Fat is other compound that influence the viscosity increase and takes place in homogenization and ageing process. During the homogenization large fat droplets are elongated and broken up into a fine emulsion of much smaller droplets, greatly increasing the surface area of the fat. Consequently, during the ageing process different amphipathic compounds are absorbed to the bare surface of the fat droplets, for example, the proteins are mostly adsorbed on the aqueous side of fat-matrix interface, with hydrophobic parts at the interface. During this part of the process the viscosity of the mix ice cream increase considerably.

Gummy is other attribute that was identified in ice cream and it is consider as defect. Under some conditions of temperature and manipulation during intake, it take a chewable texture that seems putty-like. Usually high resistance to melting has a correlation with gummy body and generally it is associated with an excessive use of stabilizers (Marshall et al., 2003; Soukoulis et al., 2010; Varela et al., 2014). A dense texture is an attribute that could be confused with gummy. Nevertheless, this defect is associated with high solids content of the mix, especially increased fat and sugar. Other suggested causes are too much stabilizer, and/or a low overrun (Guinard et al., 1997).

Creaminess is a sensory attribute that is perceived through a complex pattern such as thickness, wateriness, friction, coarseness, mouthcoating etc. Creaminess is inversely related to hardness. When ice cream has a soft texture, the hardness values are low. Generally, the hardness of ice cream is due to the free water availability which influence the formation of ice crystals during the manufacture. Both, ice crystals and ice phase volume determine the ice cream hardness, but large ice crystals affect more the texture (Goff, 1997). Hardness is inversely related to fat and solid contents (Guinard et al., 1997; Roland et al., 1999; El-Nagar et al., 2002). Following the same reasoning, an inverse relationship between hardness and overrun has been reported, since during the whipping step the partial destabilization of milk fat leads to the formation of fat networks which stabilize the ice cells. Thus, ice cream with better foamy and smooth textures (Goff, 1997; Sakurai et al., 1996; Muse and Hartel, 2004; Sofjan and Hartel, 2004). Probably that is the reason why Santa clara, Häagen-Dazs and Nestlé (Figure 2 b) were characterized as Frothy texture. It is knowing that milk fat has been recognize as very important compound for the formation and support of ice cream structure as well as for the perceived textural quality e.g. lubrication of tongue, increase of mouth coating effect, enhancement of creaminess, thickness and flavor perception. The increase of milk fat improves many attributes as creamy and greasy and decrease of hard, coarse, watery and brittle (de Wijk et al., 2006; Dresselhuis et al., 2008; Granger et al., 2005; Hyvönen et al., 2003). On the other hand, the use of hydrocolloids affects both, the texture and flavor perception of ice cream throughout different mechanisms as: control of recrystallization phenomena, viscosity increase and water retention, stabilization, emulsification and volatile aroma compounds entrapment. It had been reported that add 0.3-0.4% of hydrocolloids prevent coarseness and decrease hardness. Creaminess ice cream samples were characterized when were used intermediate percentage of hydrocolloids (Regand and Goff, 2003).

4.1.3. Flavor and residual sensation

Some samples were related to vanilla and caramel odor and flavor, such is the case of normal control Deslice, Vital´ice, Precissimo, Nestlé were marked with odor and flavor vanilla (Figures 1 and 2 c). Häagen-Dazs was characterized only as vanilla flavor (Figure 2 c). It is important to mention that the price of these ice creams are high compared to brands like the Michiocana or Valley Food which were mentioned as caramel odor and flavor, sweet and after taste. Fats has a very important function on the perception of different flavors, due to fat act as solvent reservoir, slowly releasing volatiles flavor during the consumption in ice cream (Chung and Grün, 2003). As we have mentioned previously, the milk fat is an important compound that while increase vanilla, sweet and milky attributes were enhanced whereas sour, bitter and astringent attributes were depressed due to the dissolution of lipophilic vanillin in the polar fat substrate favored the temporary control of vanilla flavor release (Hyvönen et al., 2003). On the other hand, sweetness ratings increased with sugar content and higher vanilla, almond, butery, custard/eggy, sweetness, fatty, creamy, doughy and mouth coating characteristics was found (Guinard et al., 1997). The salty flavors that appeared in low concentrations in prototypes LF and LFS (Figures 2 c) may arise from the non-fat solids, principally if were use whey powder due to the natural milk salts. Nevertheless, this off-flavors are suppressed by sweetness.

The attribute artificial aftertaste probably is due to the addition of vanilla flavor (types and intensity) that in some samples is perceived as “unnatural flavoring” which in ice cream may convey the sensation of being too high in flavoring. The synthetic or imitation vanilla that is add with vanilla extracts, may tend to produce a burning sensation on the sides and base of the tongue. Frequently, the flavor aftertaste is found in ice creams that are labeled as “vanilla flavored” or “artificially flavored vanilla” (Alvarez, 2009).

4.2. TDS and TCATA discussion

4.2.1. Vanilla, sweet flavors and artificial aftertaste

Ice cream is a product extremely sweet in comparison with other dairy products. For the assessors this a little problem due to the difficult to quantified of identified sweetness or other flavors notes. Other obstacle that the ice cream assessors has is simply taste bud fatigue due to the combined effect of sweetness and coldness on the organs of taste. Additional difficult for the ice cream judge is the mouth coating effect of milkfat. Some of the taste bud sites may be partially coated or blocked by milkfat, and hence lessen the ease of taste perception (Guinard et al., 1997).

It was observed how in spite of fat and sugar decrease (LF and LFS prototypes) all the samples showed as dominant in vanilla and sweet flavors, it was using TDS methodology (Figure 3 a-c). By other hand, TCATA analysis showed that all the samples (Figure 4 a-d) were characterized by sweet flavor but in different duration in the time of eating period. As well normal control (Figure 4 a) was characterized by vanilla flavor and fatty sensation. The prototype LFS (Figure 4 c) was described as vanilla flavor. Probably, the use of agave fructans mimic the fat and sugar functionality. There is not much knowledge of agave fructans about psychochemical and sensory properties in different foods. In general, oligofructose in the pure form has a sweetness of about 35% in comparison with sucrose. There are studies about the comparison of blue agave fructans and others natural syrups when in agave fructans was found abundance of fructose, shortage of glucose and apparent absence of sucrose (Mojica and Pérez, 2013). In low-fat yogurt the use of 6.0% of agave fructans improved sensory attributes such as flavor, viscosity, creaminess and overall acceptance compared with full fat control (26 g of milk fat). The branched structure of agave fructans may confer different functional properties than those reported for lineal fructans of inulins, giving way to new technological applications (Crispìn et al., 2015). It has been reported the benefits of agave fructans as natural prebiotic, dietary fiber and their technological functions (stabilizer, sweetener, moisturizer, gelling etc.). Thus, considering that agave fructas have demonstrated higher water absorption capacity compared with chicory inulin (fructans with lineal structure) and act as stabilizer, we could compare the agave fructans effect with hydrocolloids. it has been demonstrated that low concentrations of hydrocolloids enhance vanilla and sweet attributes and astringency constraint and thus to depression of yogurt volatile compounds release (Soukoulis et al., 2015). Normal control (Figure 3 a) showed dominance in artificial aftertaste that lasted while eating ice cream. As mentioned early, the “unnatural flavoring” could be due to the addition of synthetic or imitation vanilla extracts (Alvarez, 2009).

4.2.2. Fatty sensation

The assessors could have noticed this attribute by the presence of butter particles in the mouth after the ice cream has melted, or by a distinct greasy coating of the mouth surface after expectoration. The fat globule should have a size less than about 30–50 mm, since visible fat particles form in the samples with the associated buttery defect (Eisner et al., 2004). In a previous investigation, the use of stabilizers (egg, cream, hydrocolloids) ended up a viscous liquid that implanted a perception of mouth coating as dominan in vanilla ice cream samples (Varela et al., 2014).

The prototype LF (Figure 3 c) showed significant dominance of fatty sensation during the period of consumption when was used TDS methodology. In this low butyric fat ice cream was use agave fructans as replacement (3.0%). In previous studies, we have studied the effect of chicory inulin in low buttiric and vegetable fat ice cream (Pintor et al., 2013) as well as the effect of agave fructans on low fat and sugar ice cream (Pintor et al., 2017, and for both works the use of 3.0% improve overrun, apparent viscosity, texture and melting properties. Probably, the fat sensation is due to an increase in creaminess of ice cream that could be provoked by the decrease of free water (caused by the agave fructans and water binding) which is related to the reduction of ice crystals. Viscosity is other parameter that could be modified due to the interaction between the agave fructans and water, which is affected by both, the contribution of soluble solids to the aqueous phase and by the water-binding effect of fructans which form a gel-like network that modify the viscosity mix. Some fat and sugar replacers as inulins have been employed to increase viscosity in reduced fat ice cream (Aime et al., 2001; Aykan et al., 2008; Karaca et al., 2009).

4.2.3. Ice crystals

This attribute is considering a common texture defect in ice cream and it may be characterized by the large ice particles that provoke feeling of coldness with in the mouth, hardness and it frequently is related to rough visual appearance. The strong direct relationship between ice crystals and development of coarse and/or icy texture is well known (Regand & Goff, 2006). In contrast, when the structure of the ice cream is well stabilized the ice crystals remain small. Since finer structures, in general, produce sensory properties such as creaminess and smoothness, coldness has less impact in these samples. Formation of ice crystals plays an essential role in determining the final quality of ice cream, and small crystal sizes are desirable (Adapa et al., 2000). Other important aspects are the temperature and the speed to freeze the ice cream due to the ice crystals tend to increase in size when storage time are extended. An effect called ”nucleation” arises during this period; the larger crystals become larger at expense of the small ice crystals, which disappear and over time the ice cream become coarser. The way to perceive this characteristic is put a little some sample in the mouth and bite between the upper and lower incisors, observing the resistance to come together. Other technique is pressing the sample of ice cream between the tongue and palate. Lately, the ice cream industry is focus on freezing and storage technology to produce small ice crystals and delay their growth during storage or distribution caused by temperatures fluctuations. In previously thermal investigations, the use of 3.0% of agave fructans promoted a positive effect on thermal properties in low fat and low fat and sugar ice creams, resulted in the highest percentage of frozen water, reducing free water, which can undergo possible state transitions such as ice crystallization (Pintor et al., 2018). Apparently, agave fructans had the ability to reduce the number and formation of ice crystals due to both, the influence of soluble solids in the aqueous phase and the water-binding effect of fructans. In this work, the prototype LFS (Figure 3 d) showed ice crystals as dominant attribute. Probably, the use of only 3.0% of agave fructans difficult the compensation of both, the fat and sugar. On the one hand, the compensation of solids caused by fat and by other hand the decrease of the freezing point produced by sugar.

TDS and TCATA methodology are dynamic sensory test that have been studied by many authors to know the advantage and disadvantage between each one (Ares et al., 2015;2016; Jaeger et al., 2017; Pineu et al., 2012; Nguyen et al., 2018). The principal differences between the methodologies are the number of selected attributes at each moment of evaluation. For TDS only is selected the dominant attribute, while in TCATA can be selected all the attributes that describe food in each moment of the evaluation. If the TDS and TCATA results are compared, it was observed that some attributes marked as significant for TDS were not for TCATA results. It was also observed that more attributes were dominants for each sample in TDS methodology in comparison with TCATA. For example, for La Michoacana sample the dominant attributes were vanilla and sweet flavor and fatty sensation while TCATA only sweet flavor and fatty sensation were significant (with different duration in the time of eating period). This means that as dominant attributes they had a greater impact in assessor perception that as attributes that describes the sample. The selection of TCATA attributes does not mean that the assessors only discriminated the attributes from attributes list, rather they had the enough citation to be consider significant from the rest.

In different studies Ares et al. (2015 and 2016) found that TDS and TCATA showed similar results which agrees with this investigation. Consistently, the attributes frequently used in TCATA methods were denominated as dominant attributes for TDS, suggesting that the attributes that most attracted attention to be marked as dominant were relevant to describe the samples. In the same investigation was found how TCATA showed detail result more significant than TDS due to two important aspects: the first one is the process of the method and the second one is the assessor criteria about the "dominant attribute". Labbe et al. (2009) define "dominant" as the most striking sensation which it is not necessarily the most intense. In this study we came to a different analysis since for TDS the results were more detail for each dominant attribute as well as the intensity of each one and the moment of the decrease or disappear of the perception of assessors. According to the methodology followed and the bibliography consulted, it suggests that the judges, having already a previous training for the test of the sensory profiles that involved training with standards, calibration and re-training in different tests, had a better understanding of the attributes and how to measure them, as well as the same definition of dominant attribute. Another possible cause could be the presentation of the attributes in the questionnaire, since in TDS they were shown in two parts, separating flavor attributes on one side and texture attributes on the other, this in order to concentrate them in an independent and able to take more sample between each one. This method had been reposted as M-TDS or TDS by modality for Agudelo et al. (2015.). However, for TCATA all the attributes were shown simultaneously on a single screen so that they could select all the attributes they considered relevant to describe the sample at each consumption time. Causing the evaluation with less attention separately at the same time. Ares, et al, (2015), suggests that TDS seems to be the most appropriate method when the research question requires the identification of the attributes that capture the attention of the judges at each moment of the evaluation.

5. Conclusions

A conventional descriptive analysis was development to understand the importance of the ice cream formulation especially when indispensable compound as fat and sugar are reduced. In general, samples with milk fat or butyric fat were characterized with the better appearance, texture, odor and flavor attributes being the ice cream with desirables characteristics. The reduction of sugar was reflected especially in undesirable textures. By other hand, agave fructans had a better functionality in low fat ice creams when were compared with a normal control. Nevertheless, in low fat and sugar samples, it did not compensate at all, due to attributes as hardness and ice crystals appeared.

The use of agave fructans as fat and sugar replacer in ice cream prototypes produced some differences between dominant attributes when we use two dynamic methodologies. For both methods, TDS and TCATA, vanilla and sweet flavors were the most perceptible attributes during the eating period when 3.0% of agave fructans were used reduced fat and sugar ice cream prototypes. In TDS test, the artificial aftertaste appeared later in the consumption, probably for the addition of synthetic or imitation vanilla extracts.

Fatty sensation was other dominant attribute in LF prototype detected during TDS method, probably this sensation was provoked by the increase of creaminess caused by the decrease of free water (produced by the agave fructans and water binding) which is related to the reduction of ice crystals.

Ice crystals was an attribute predominate during ice cream consumption especially when fat and sugar were reduced. Possibly, for agave fructans is difficult to compensate both fat solids and the decrease of freezing point caused by sugar. Therefore, It is proposed to increase the agave fructans concentration in ice cream prototypes to avoid the grow ice crystals and enhance texture.

The use of TDS and TCATA can obtain important results, but the propose of use are different. For example, TDS provides a significant description of attributes when the researcher is interested in one attribute at an especial time. In this work, we obtained a better significant when the attributes were presented in two steps (this method had been investigated and it has been called M-TDS). Other possibility could be the exhaustive training of assessors which resulted in a better understanding of the attributes and how to measure them, as well as the same definition of dominant attribute. TCATA resulted an important tool when you want to know information about the interaction between the whole set of attributes, enabling to represent more than two attributes at any point of consumption time.

6. Acknowledgments

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8. Conclusiones generales

El helado es un producto muy complejo en cuanto a su microestructura, la cual está influenciada directamente por la cantidad y calidad de sus ingredientes, así como cada uno de los pasos del proceso de manufactura. La modificación de la formulación trae consigo diversos retos tecnológicos para los fabricantes, tanto sensorial, térmica y texturalmente hablando. La reducción de grasa y azúcar en helados, abre camino a nuevos compuestos que imitan o remplazan a estos ingredientes tan importantes, mejorando sus propiedades y potencializando su consumo.

El uso de inulina de achicoria en helados reducidos en grasa y azúcar mejoraron las propiedades de textura, resultando en valores de viscosidad aparente y *overrun* mayores en comparación con las muestras que no los contenían, lo que sugiere que la inulina tuvo la capacidad de controlar la disposición de agua, mostrando un sistema más estable. También mejoró las propiedades de derretimiento provocando tiempos de fusión más grandes, lo que se traduce en una matriz estable, en donde cristales de hielo, grasa emulsionada y burbujas de aire se encuentran en equilibrio. Por otro lado, la textura instrumental fue afectada positivamente por la inulina debido a que este compuesto tuvo la habilidad de retener agua libre cuando grasa y azúcar fueron reducidas, dando como resultado menor cantidad y tamaño de cristales de hielo, lo que se vio reflejado en texturas más suaves. En las condiciones experimentales propuestas, la inulina de achicoria pudo ser empleada para reducir hasta un 30% del contenido de grasa butírica y 12% de azúcar.

En la segunda publicación se estudió el efecto de fructanos de agave en dos formulaciones, helados reducidos en grasa (LF) y helados reducidos en grasa y azúcar (LFS) sobre las propiedades térmicas. Dando como resultado, que el uso de aproximadamente 3.0 g/100 ml de fructanos de agave redujo la cantidad de agua libre y por lo tanto incrementó la cantidad de agua ligada. Lo que se vio reflejado en las temperaturas de transición vítrea y de fusión, las cuales fueron afectadas positivamente. Por otro lado, el uso de espectrofotometría infrarroja mostró un incremento en la magnitud de diferentes bandas, especialmente en los grupos O-H que corresponden al hidrogeno polimérico causado por la interacción de fructanos de agave y agua.

En la tercera publicación, con base a una triple correlación (sensorial, térmico y textural), en helados bajos en grasa y bajos en grasa y azúcar, usando fructanos de agave como remplazo, atributos sensoriales como la textura y aspecto cristalizado, textura dura y granulada, sensación de frío, entre otros, se relacionaron con las formulaciones que no contenían o estaban contenidas en una concentración menor al 1.2% de fructanos de agave. Al mismo tiempo, estas formulaciones se correlacionaron con propiedades térmicas como el agua congelada, la fracción de hielo, la entalpía y las propiedades de temperatura de fusión, así como propiedades de textura, dureza, velocidad de fusión y fuerza de compresión. Aparentemente, la baja concentración de fructanos de agave promovió el incremento de cristales de hielo que causaron formulaciones de helado con texturas duras. Por otro lado, las muestras con concentraciones de 1.2 a 3.0% de fructanos de agave helado con largos tiempos de fusión, texturas suaves, cremosa, fluida y sensación a grasa. Estas propiedades se relacionaron directamente con las propiedades de viscosidad aparente y valores de *overrun*, así como con bajas concentraciones de agua no congelada, altas temperaturas de transición vítrea y valores de ΔCp.

Con base en el estudio de agrado, las muestras que más gustaron a los evaluadores fueron las que contenían todos los compuestos, aunque todas las muestras tenían una puntuación promedio de agrado en la escala hedónica.

Finalmente, en la cuarta publicación se mostraron los resultados del análisis descriptivo para muestras comerciales de helado y muestras prototipo (contenidas con fructanos de agave). Dando como resultado que en general las muestras contenidas con grasa de leche se caracterizaron con los mejores atributos relacionados a la textura, apariencia, olor y sabor. Los fructanos tuvieron un mejor efecto en el prototipo reducido en grasa (LF) en comparación con los helados reducidos en grasa y azúcar (LFS), que mostraron características indeseables como dureza y cristales de hielo (Perfiles descriptivos se muestran en el Anexo 1)

De acuerdo con las gráficas TDS, el uso de aproximadamente de 3.0% de fructanos de agave en los helados prototipo LF compensaron dulzor, sabor vainilla y sensación grasa, debido a que estos atributos fueron más dominantes durante el tiempo de consumo. Lo que sugiere que los fructanos tuvieron la capacidad de suplir la cantidad de grasa reducida. Sin embargo, en helados reducidos en grasa y azúcar (LFS), los atributos dominantes fueron cristales de hielo, sabor vainilla y dulzor, lo que indica que probablemente los fructanos no tuvieron la capacidad de suplir las propiedades que otorgan la grasa y el azúcar que fueron eliminadas de la formulación. Al ser evaluadas las mismas muestras por la metodología TCATA se observó que el uso de fructanos de agave compensaron para ambas formulaciones (LFS y LFS) los atributos de dulzor, vainilla y sensación grasa.

Se espera que esta tesis sirva como base a otros estudios cuando compuestos tan importantes como grasa y azúcar son reducidos o sustituidos por otros compuestos en la formulación de helados.

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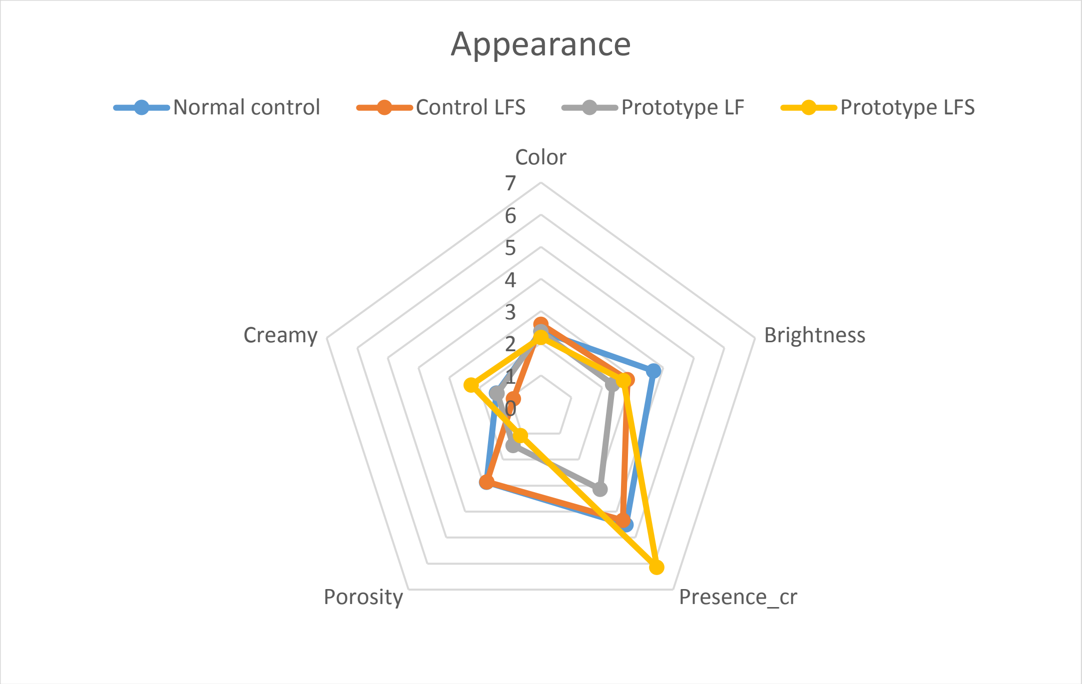
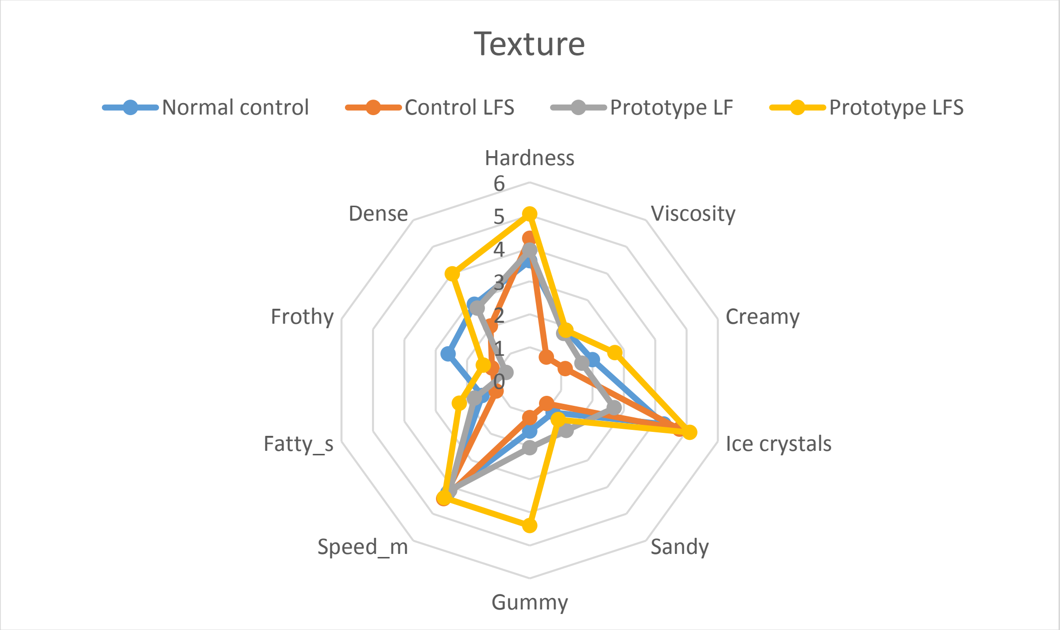
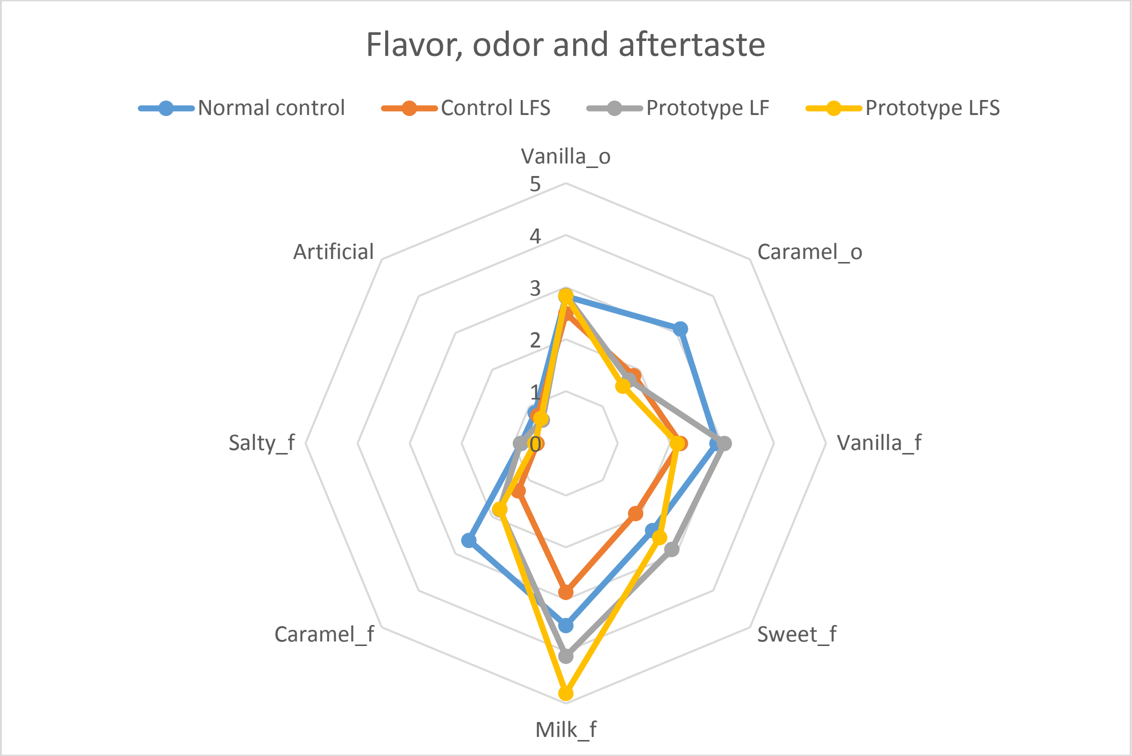
10. Anexos

Anexo 1. Spider graphic about appearance attributes.



Anexo 2. Spider graphic about texture attributes.





Anexo 3. Spider graphic about flavor, odor and aftertaste attributes.

El jurado designado por la

División de Ciencias Biológicas y de la Salud de la Unidad Iztapalapa aprobó la tesis

ESTUDIO DE PROPIEDADES FISICOQUÍMICAS, TÉRMICAS Y SENSORIALES EN HELADOS REDUCIDOS EN GRASA Y AZÚCAR USANDO INULINA DE ACHICORIA Y FRUCTANOS DE AGAVE COMO REMPLAZO

que presentó

María Aurora Pintor Jardines

Comité Tutoral:

Director: Dr. Héctor B. Escalona-Buendía (UAMI)

Asesor: Dra. Patricia Severiano Pérez (UNAM)

Jurado:

Presidente: Gabriela Mariana Rodríguez Serrano Gabriela Rodríguez

Secretario: Angélica Román Guerrero

Vocal: Patricia Severiano Pérez

Vocal: María del Carmen Hernández Jaimes

Casa abierta al tiempo
UNIVERSIDAD AUTÓNOMA METROPOLITANA

ACTA DE DISERTACIÓN PÚBLICA

No. 00216
Matrícula: 2151800101

ESTUDIO DE PROPIEDADES FISICOQUÍMICAS, TERMICAS Y SENSORIALES EN HELADOS REDUCIDOS EN GRASA Y AZÚCAR USANDO INULINA DE ACHICORIA Y FRUCTANOS DE AGAVE COMO REMPLAZO.

En la Ciudad de México, se presentaron a las 15:00 horas del día 19 del mes de julio del año 2019 en la Unidad Iztapalapa de la Universidad Autónoma Metropolitana, los suscritos miembros del jurado:

DRA. GABRIELA MARIANA RODRIGUEZ SERRANO
DRA. PATRICIA SEVERIANO PEREZ
DRA. MARIA DEL CARMEN HERNANDEZ JAIMES
DRA. ANGELICA ROMAN GUERRERO

Bajo la Presidencia de la primera y con carácter de Secretaria la última, se reunieron a la presentación de la Disertación Pública cuya denominación aparece al margen, para la obtención del grado de:

DOCTORA EN BIOTECNOLOGIA

DE: MARIA AURORA PINTOR JARDINES

y de acuerdo con el artículo 78 fracción IV del Reglamento de Estudios Superiores de la Universidad Autónoma Metropolitana, los miembros del jurado resolvieron:

Aprobar

Acto continuo, la presidenta del jurado comunicó a la interesada el resultado de la evaluación y, en caso aprobatorio, le fue tomada la protesta.

MARIA AURORA PINTOR JARDINES
ALUMNA

REVISÓ

MTRA. ROSALIA SERRANO DE LA PAZ
DIRECTORA DE SISTEMAS ESCOLARES

DIRECTORA DE LA DIVISIÓN DE CBS

DRA. SARA LUCIA CAMARGO RICALDE

PRESIDENTA

DRA. GABRIELA MARIANA RODRIGUEZ SERRANO

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