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Tipos de doce de leite e seus processos produtivos abrangendo aspectos de qualidade e inovação tecnológica

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O presente trabalho em nível de mestrado foi avaliado e aprovado por banca examinadora composta pelos seguintes membros:

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Florianópolis, 2021.

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RESUMO

O doce de leite é um produto lácteo obtido por aquecimento de leite e açúcar em condições controladas. A legislação aborda alguns demais componentes que são permitidos adição, porém alterando as denominações deste produto. Alguns adjuvantes de processo podem ser citados, como o bicarbonato de sódio para o processo produtivo, corantes, aromatizantes e conservantes. Os padrões de identidade e qualidade presentes na legislação também regulamentam parâmetros físico-químicos e microbiológicos, que são de extrema importância para evitar fraudes no produto. Seu consumo e disseminação é observado principalmente nos países da América Latina, principalmente Argentina, Brasil e Uruguai, desta forma a legislação entre os países do Mercosul são harmonizadas, facilitando processos de exportação e importação. Existe alguma disseminação deste produto também na América do Norte e na Europa (Espanha e França). Os diferentes adjuvantes de processo utilizados geram algumas variações do mesmo produto, porém com aplicações diferentes, como o doce de leite para utilização em sorvetes e confeitaria. O processo produtivo também gera uma variação de produto, principalmente pela influencia dos parâmetros sobre as reações de escurecimento não enzimático (Maillard e Caramelização), gerando consequentemente compostos químicos que podem tanto beneficiar o produto com aromas quanto denegrir a qualidade devido desenvolvimento de compostos indesejáveis (HMF) ou sabores indesejáveis. Este produto tem três formas principais de produção com algumas variações menos comuns. O processo produtivo em batelada (tacho aberto) é o mais utilizado por ser relatado como o método produtivo que provém o produto de melhor qualidade. O método de produção contínuo costuma ser mais utilizado para produção de doce de leite para confeitaria. Mesmo com o controle do processo produtivo, defeitos podem ser observados neste produto, alguns podem ser controlados e evitados como sinerese e doce de leite com escurecimento excessivo, enquanto a cristalização da lactose não pode ser evitada, apenas reduzida e esforços são feitos para reduzir o máximo este defeito. Algumas inovações tecnológicas foram observadas buscando diferentes resultados e públicos para consumo de novos produtos similares ao doce de leite. Foram escolhidas cinco palavras específicas (*Dulce de leche*; Doce de leite; *Milk jam*; *Arequipe* e Manjar blanco) para recuperação de maior número de artigos possível, bem como utilizou-se diversos bancos de dados (Science Direct, Springer, Wiley, Scielo, Scopus, Portal Capes, Taylor e Francis e Google Acadêmico). A busca na literatura por este assunto específico demonstrou um quantidade escassa de dados, levando os autores a inferir que este produto é um potencial produto para inovação e que o mesmo já possuí certo potencial devido suas características (processo produtivo robusto e bem estabelecido, além de não utilizar cadeia de frio) e sabores e aromas agradaveis.

Palavras-chave: Produto lácteo concentrado, Doce de leite, Inovação, *Dulce de leche*.

ABSTRACT

Dulce de leche is a dairy product obtained by heating milk and sugar under controlled conditions. The legislation discusses some other components that are allowed to be added, but changing the designations of this product. Some processing adjuvants can be mentioned, such as sodium bicarbonate for the production process, coloring, flavoring, and preservatives. The identity and quality standards present in the legislation also regulate physicochemical and microbiological parameters, which are extremely important to avoid product fraud. Its consumption and dissemination is observed mainly in Latin American countries, especially Argentina, Brazil, and Uruguay, so the legislation among the Mercosur countries are harmonized, facilitating export and import processes. There is also some dissemination of this product in North America and Europe (Spain and France). The different process adjuvants used generate some versions of the same product, but with different applications, such as *dulce de leche* for use in ice cream and confectionery. The production process also generates a product variation, mainly due to the influence of the parameters on the non-enzymatic browning reactions (Maillard and Caramelization), consequently generating chemical compounds that can either benefit the product with aromas or harm the quality due to the development of undesirable compounds (HMF) or undesirable flavors. This product has three main forms of production with some minor variations. The batch (open pan) production process is the most widely used as it is reported to provide the best quality product. The continuous production method is most commonly used for the production of *dulce de leche* for confectionery. Even with the control of the production process, defects can be observed in this product, some can be controlled and avoided such as syneresis and excessive browning, on the other hand, lactose crystallization cannot be avoided, only reduced and efforts are made to reduce this defect as much as possible. Some technological innovations were observed seeking different results and target consumers for consumption of new products similar to *dulce de leche*. Five specific words were chosen (*Dulce de leche*; Doce de leite; *Milk jam*; *Arequipe* and Manjar blanco) to retrieve as many articles as possible, and several databases were used (Science Direct, Springer, Wiley, Scielo, Scopus, Portal Capes, Taylor and Francis and Google Academic). The literature search for this specific subject showed a scarce amount of data, leading the authors to infer that this product is a potential product for innovation and that it already has some potential due to its characteristics (robust and well-established production process, besides not using cold chain) and pleasant flavors and aromas.

Keywords: Concentrate dairy product, Doce de leite, Innovation, *Dulce de leche*

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LISTA DE ABREVIATURAS E SIGLAS

CIELAB Espaço de Cor Utilizado na Determinação de Cores

GMP Boas Práticas de Fabricação

HMF Hidroximetilfurfural

IBGE Instituto Brasileiro de Geografia e Estatística

IJDT International Journal of Dairy Technology

INDEC Instituto Nacional de Estatística e Censos da Argentina

INS Sistema Internacional de Numeração de Aditivos

MAPA Ministério da Agricultura, Pecuária e Abastecimento

MERCOSUL Mercado Comum do Sul

MGAP Ministério da Pecuária, Agricultura e Pesca (Argentina)

PIA Pesquisa Industrial Anual

SPME-GC-MS Microextração em Fase Sólida – Cromatografia Gasosa – Espectometria de Massa

SUMÁRIO

1 INTRODUÇÃO

O doce de leite é um produto típico da América Latina, produzido a partir da mistura de leite e açúcar, aquecido sob condições controladas, gerando um produto com cor semelhante à de caramelo e sabor agradavelmente doce. Pode ser consumido vertendo-se em pães e biscoitos, com frutas e queijos e é utilizado em produtos de padaria, sorvetes e confeitaria (MERCOSUL, 1996; HYNES; ZALAZAR, 2009; SILVA *et al*., 2015). O doce de leite é mais vendido e consumido na Argentina, no Uruguai e no Brasil, mas demais países da América Latina como México e Cuba também possuem consumo destacável. Segundo o país onde é produzido e comercializado, este produto apresenta denominações diferentes e pode ter algumas diferenças em sua composição ou processo de produção. No México, por exemplo, este é produzido principalmente a partir do leite de cabra. Em Cuba, o processo de produção resulta em um produto final com menor umidade, sendo normalmente vendido em pequenas barras ou tabletes (PENCI; MARÍN, 2016).

No processo de produção do doce de leite, o leite e o açúcar são aquecidos sob condições controladas, o que leva a dois efeitos principais: evaporação da água e consequentemente concentração de sólidos, além disso, o bicarbonato de sódio também pode ser utilizado, influenciando positivamente no processo produtivo (ZALAZAR; PEROTTI, 2011). O tempo e a temperatura em que o leite é aquecido influenciam a reação de Maillard, a caramelização dos açúcares e, consequentemente, as propriedades do produto final (GAZE *et al*., 2015).

A lei do Mercado Comum do Sul (Mercosul) define as características, classificação e requisitos de qualidade para o doce de leite comercializado em seus países membros (Argentina, Brasil, Paraguai e Uruguai). O Brasil tem sua própria legislação harmonizada com as determinações do Mercosul (MERCOSUL, 1996; MAPA, 1997). Diferentes tipos de doce de leite são descritos pela legislação do Mercosul sob diferentes categorias: doce de leite em pasta, o tipo mais comum, com um valor máximo de umidade de 30% (p/p); doce de leite para ser aplicado em sorvetes; doce de leite adicionado de outros ingredientes; doce de leite para a indústria de confeitaria; e doce de leite adicionado de creme (MERCOSUL, 1996; ZALAZAR; PEROTTI, 2011). Os diferentes tipos de doce de leite encontrados no mercado são o resultado de variações nos parâmetros do processo de produção. Três principais processos de produção são mencionados na literatura: batelada, semi-contínuo e contínuo (ZALAZAR; PEROTTI, 2011).

Em relação às inovações, vários estudos foram desenvolvidos com diferentes propósitos, tais como a substituição de parte da fração lipídica a fim de alterar o perfil lipídico (RANALLI; ANDRÉS; CALIFANO, 2017a; CHAVES *et al*., 2018); a substituição de açúcar por adoçantes ou prebióticos (GUIMARÃES *et al.*, 2012; CARDOSO *et al*., 2018), como por exemplo, o estudo realizado por Leddomado *et al*. (2021) que empregaram os prebióticos inulina e oligofrutose; e o desenvolvimento de produtos utilizando leite de outros animais que visou aumentar a versatilidade dos sabores, além de agregar valor (WANDERLEY *et al*., 2005; CHAVES *et al*., 2018).

O doce de leite, sendo um produto lácteo, tem notável valor nutricional e, embora não seja bem conhecido fora da América Latina, alguns países da Europa (Espanha e França) já começaram a comercializar este produto (ZALAZAR; PEROTTI, 2011; PENCI; MARÍN, 2016). As características de aroma, sabor e tecnologia de produção tornam o doce de leite um produto interessante a ser estudado. Estudos sobre o doce de leite devem ser incentivados, a fim de aumentar a divulgação e comercialização de um produto com diversos atributos sensoriais.

2 OBJETIVOS

2.1 OBJETIVO GERAL

Este trabalho teve como objetivo geral transmitir informações sobre o doce de leite, buscando sua difusão tanto na forma cientifica quanto popular devido seus sabores e características únicas.

2.1.1 Objetivos Específicos

- Indicar as principais características de produção do doce de leite; Descrever os métodos mais comuns de produção e os mais utilizados na industrialização;
- Descrever os principais defeitos que ocorrem neste produto e as formas de evitar estas falhas na qualidade;
- Descrever os principais ingredientes e as suas influências nas características do produto final;
- Abordar inovações no que diz respeito à novos ingredientes, produtos e formas de produção do doce de leite;
- Abordar dados produtivos deste produto; Demonstrar como este se comporta perante o mercado nacional e internacional;
- Compartilhar as informações legislativas deste produto; Demonstrar leis que regulamentam os padrões de identidade e qualidade do doce de leite;
- Descrever os tipos de doce leite encontrados no mercado conforme aplicação que será utilizado;
- Por fim, disseminar informações técnicas e produtivas deste produto, instigando seu consumo e levar a indústria conhecimentos científicos que gerem inovações no processo produtivo e nos produtos relacionados ao doce de leite.

3 REVISÃO BIBLIOGRÁFICA

3.1 DEFINITIONS OF THE *DULCE DE LECHE*

Dulce de leche is defined as the product obtained by concentration of milk by heating at normal or reduced pressure, with the addition of sucrose and other ingredients (MERCOSUL, 1996; MAPA, 1997; GAZE *et al*., 2015).

The legislation for *dulce de leche* has an agreement between Mercosul countries in order to standardize the production parameters (mandatory ingredients, optional additives and trade denominations) and the characterization parameters (physicochemical and microbiological), seeking to facilitate the trade between these countries (ZARPELON *et al*., 2016).

This product has different denominations according to the countries where it is marketed, being called *dulce de leche* in Argentina and Uruguay and doce de leite in Brazil. In Mexico it is made from goats' milk and is known as *cajeta*. In Panama, Venezuela and Colombia it is entitled "*arequipe*". Manjar blanco is the denomination used in Peru, Chile, Ecuador, and Bolivia. In Cuba, it is called *dulce de leche cortada*, being a solid and usually sold as small bars or tablets (GAZE *et al*., 2015; PENCI; MARÍN, 2016).

3.2 PRODUCTION DATA OF THE *DULCE DE LECHE*

The *dulce de leche* produced in Latin America is commonly exported to the European Union and the United States (RANALLI; ANDRÉS; CALIFANO, 2017a).

In Brazil, according to the Annual Industrial Survey (PIA) released by the Brazilian Institute of Geography and Statistics (IBGE), the production of *dulce de leche* was close to 82,624 and 74,955 tonnes for the years of 2017 and 2018, respectively. Between the 2010 and 2016, the southeast region of the country led dairy production, followed by the South (second largest producer) and Northeast (third largest producer) regions. The Northeast and Midwest region have limited production (GOMES *et al*., 2017).

Regarding export values, from January to August 2016, the Brazilian state of Minas Gerais exported more than US$ 106,000 in *dulce de leche* to countries such as the United States, United Kingdom, Angola, Bolivia, among others. With these values in mind, the state of Minas Gerais is currently the first place in the national ranking, followed by São Paulo,

with US$ 27,000 and Santa Catarina, with US$ 13,000. Only in 2014, Brazil's *dulce de leche* exports were equivalent to US$ 239,000 (SEBRAE, 2015; COMEXSTAT, 2020). Considering imports, in 2016, an 82% increase was observed when compared to the previous year, with Argentina being the major country from which Brazil bought *dulce de leche* (COMEXSTAT, 2020).

According to the National Institute of Statistics and Census of Argentina (INDEC), the country produced 92,593 tonnes and 100,575 tonnes of *dulce de leche* in 2017 and 2018, respectively. In 2017, 3,331 tonnes were exported, worth over US$ 7.8 million. Among the countries that Argentina exports to, we can mention: Brazil, Canada, Chile, Japan, Syria and the United States. Regarding *dulce de leche*'s production in Uruguay, the Ministry of Livestock, Agriculture and Fisheries declared that the country produced 15,412 tonnes and 16,521 tonnes of *dulce de leche* in 2017 and 2018, respectively. In 2018, 219 tonnes were exported and *dulce de leche* was the third most produced dairy product in the country, only behind the production of powdered milk and cheese (MGAP, 2019).

3.3INGREDIENTS USED IN THE PRODUCTION OF THE *DULCE DE LECHE*

Common *dulce de leche* can be composed of milk or reconstituted milk and sucrose in a maximum value of 30 kg per 100 L of milk. The addition of milk solids and/or cream is allowed. Sucrose content could be partially replaced by monosaccharides and/or other disaccharides at a maximum value of 40 % w/w (MERCOSUL, 1996).

Milk is the main ingredient used in the production of *dulce de leche*, so the quality of this ingredient is of great importance for the final product's characteristics. The milk to be used in the manufacture of *dulce de leche* must be of good quality, as the increased acidity (caused by microorganisms) may unbalance the milk components and the presence of thermostable enzymes results in a subpar product. Also, the milk should be stable to the heat treatment necessary for *dulce de leche* production; furthermore, it is recommended that the milk possesses a high solid content, since fat is directly related to the texture of the product (STEPHANI *et al*., 2019).

To produce *dulce de leche*, the milk must be microbiologically safe and the use of milk from colostrum is not allowed. If the milk's acidity is greater than 0.20 % of lactic acid, this may be corrected with sodium bicarbonate, in a higher amount than usual, which would

be ideal to neutralize until 0.20 % lactic acid, however, this can lead to the saponification of free fatty acids and production of unpleasant flavours (ZALAZAR; PEROTTI, 2011).

Good agricultural practices, genetics, and bovine management can affect the milk's characteristics as well as that of final product, since milk is the major component of *dulce de leche*. The variability of the process parameters, such as heating time and temperature, use of increased or reduced pressure, the pressure intensity and mass balance, also affects the product's characteristics (GAZE *et al*., 2015).

As optional ingredients and process adjuvants, it is possible to mention neutralizing agents, starches, modified starches, flavorings, colorants and preservatives, among others (MERCOSUL, 1996).

To achieve the desired solids content (between 68 % and 70 %), sucrose, glucose, or both may be added. Sucrose is generally more commonly used to produce *dulce de leche* and the presence of sucrose is related to the flavors and aromas of this product, since it is part of the Maillard and caramelization reactions, which in turn, during the cascade of chemical reactions, produce chemical compounds that positively characterize the product (ROZYCKI; BUERA; PAULETTI, 2010; ZALAZAR; PEROTTI, 2011). Glucose can be added, usually in quantities below than 2 % to 5 % (w/w) in order to provide a more viscous texture, a brighter appearance, and improve the product's traditional brown color, however, it should be added near the end of the heating process as to avoid excessive browning. The addition of starch or modified starch with a maximum value of 0.5 g / 100 mL of milk is also permitted. Table 1 presents the additives and technological coadjuvants with their maximum levels, as determined by Mercosul law.

Table 1 – Additives and coadjuvants of technology and elaboration of *dulce de leche* allowed in Mercosul legislation.

Additives and coadjuvants	**Function**	**Maximum concentration**
Sorbic acid and its sodium, potassium or calcium salts	Preservative	600mg/kg (in sorbic acid) 1000mg/kg (in sorbic acid) for exclusive industrial use
Natamycin (in free surface)	Preservative	1 mg/dm2
Calcium Lactate	Texturizer	G.M.P.
Vanilla flavoring, vanillin and/or ethyl vanillin isolated or in mixtures	Flavoring	G.M.P.
Sodium citrate	Stabilizer	G.M.P.
Sorbitol	Umectant	5g/100g

Caramel (INS 150 a,b,c,d)	Corant	G.M.P.
Alginic Acid	Thickener/ Stabilizer	5000mg/kg(*)
Ammonium Alginate	Thickener/ Stabilizer	5000mg/kg(*)
Calcium Alginate	Thickener/ Stabilizer	5000mg/kg(*)
Carrageenan including furcelarana and sodium and potassium salts	Thickener/ Stabilizer	5000mg/kg(*)
Pectin and Pectin-starch	Thickener/ Stabilizer	5000mg/kg(*)
Potassium Alginate	Thickener/ Stabilizer	5000mg/kg(*)
Propylene glycol alginate	Thickener/ Stabilizer	5000mg/kg(*)
Sodium Alginate	Thickener/ Stabilizer	5000mg/kg(*)
Agar	Thickener/ Stabilizer	5000mg/kg(*)
Carboxymethyl cellulose	Thickener/ Stabilizer	5000mg/kg(*)
Sodium carboxymethyl cellulose	Thickener/ Stabilizer	5000mg/kg(*)
Methyl cellulose	Thickener/ Stabilizer	5000mg/kg(*)
Methyl ethyl cellulose	Thickener/ Stabilizer	5000mg/kg(*)
Hydroxypropyl cellulose	Thickener/ Stabilizer	5000mg/kg(*)
Arabica Gum	Thickener/ Stabilizer	5000mg/kg(*)
Xanthan Gum	Thickener/ Stabilizer	5000mg/kg(*)
Garrofin Gum	Thickener/ Stabilizer	5000mg/kg(*)
Karaya Gum	Thickener/ Stabilizer	5000mg/kg(*)
Gellan Gum	Thickener/ Stabilizer	5000mg/kg(*)
Tragacanth Gum	Thickener/ Stabilizer	5000mg/kg(*)
Konjak Gum	Thickener/ Stabilizer	5000mg/kg(*)
Gelatin	Thickener/ Stabilizer	5000mg/kg(*)
Microcrystalline cellulose	Thickener/ Stabilizer	5000mg/kg(*)
Betagalactosidase	Lactase	G.M.P.
Sodium bicarbonate	Browning	G.M.P.
Sodium hydroxide	Browning	G.M.P.
Calcium hydroxide	Browning	G.M.P.
Sodium carbonate	Browning	G.M.P.

(*) Use of these stabilizers/thickeners in a mixture should not be more than 20,000 mg/kg of the final product.
G.M.P. means Good Manufacturing Practices.
INS means International Numbering System and is used for the identification of food additives.
Source: Mercosul (1996).

Alkali neutralizing agents, normally sodium bicarbonate and calcium hydroxide, are generally used during the process. The purpose of this agent is to reduce the milk's acidity and to prevent destabilization of casein micelles. Acidification during the evaporation process is related an increase in colloidal calcium phosphate, the formation of organic acids from the degradation of lactose, and the breakdown of casein phosphate esters. The use of sodium bicarbonate is important to avoid defects in the final product due to the loss of casein stability due to the production process and the consequent acidification of the medium, so its application is always recommended to stabilize the medium and also due to its participation in

the non-enzymatic browning reactions. (MERCOSUL, 1996; HYNES; ZALAZAR, 2009; ZALAZAR; PEROTTI, 2011).

To enhance the aroma, vanilla, vanillin, and/or ethyl vanillin flavorings can be added to the product, according to Mercosul's legislation and good manufacturing practices. The addition occurs at the end of the *dulce de leche* process (at approximately 65 ºC) due to these compounds being heat sensitive. The addition of preservatives such as sorbic acid and its respective sodium, potassium and calcium salts are allowed in maximum values of 600 mg/ kg when added to *dulce de leche* for direct consumption and 1000 mg/kg in *dulce de leche* for industrial use (confectionery products, ice cream, and others). They are used to reduce chances of mold growth in the product (MERCOSUL, 1996; ZALAZAR; PEROTTI, 2011).

3.4 *DULCE DE LECHE* TYPES AND USES

Seeking to diversify the product, the legislation allows the addition of other ingredients, such as peanuts, cocoa, chocolate, and dried fruits. The substitution, when it occurs, must be between 5 % and 30 % (w/w) to not mischaracterize the *dulce de leche*. For *dulce de leche* with a specific use in ice cream manufacture, the addition of colorings are permitted. For use in confectionery, stabilizers, thickeners, and/or wetting agents could be added (MERCOSUL, 1996).

Dulce de leche added with dairy fat is a variation with high caloric value. However, due to market changes, such as customer's preference for products with reduced fat content and the increased price of this kind of *dulce de leche*, it has lost some of its popularity and the production of this type of *dulce de leche* is relatively scarce in large batches (MERCOSUL, 1996; PERRONE *et al*., 2017; STEPHANI *et al*., 2019).

For *dulce de leche* to be used in ice cream production the addition of coloring is permitted, as *dulce de leche* must have a pronounced color and taste in order to be noticed in the complex matrix of flavors and aromas already present in a product such as ice cream. *Dulce de leche* to be used in confectionery is produced on a large scale and sold in larger packages than common *dulce de leche*, it is also more affordable due to the presence of stabilizers, thickeners, and wetting agents. *Dulce de leche* for confectionery use needs to have a pronounced taste, high viscosity to be molded more easily and lower microbial counts than common *dulce de leche* due to its production process, using higher temperatures than the common *dulce de leche* (use of continuous production process) because the microbiological

count should be lower. This *dulce de leche* will still be added to another product which must also be microbiologically safe, i.e., the *dulce de leche* for confectionery is still manipulated for its addition to other products. (PERRONE *et al*., 2017; STEPHANI *et al*., 2019).

The production of diet *dulce de leche* requires special attention due to the reduction of sucrose, which directly influences product's sensory attributes. Sucrose influences several of the process's parameters, and when the removal of sucrose is necessary, compounds that are able to compensate for the lack of sucrose should be added, such as pectin, sorbitol, modified starches, and sweeteners. It is important to emphasize the importance of sucrose in the sweet taste of the product, but also its complex influence on the texture parameters, so when sucrose is removed from the production process, substitutes to provide the sweet taste (sorbitol, xylitol, sweeteners) and also texture agents (pectin and modified starches). (PERRONE *et al*., 2017; STEPHANI *et al*., 2019).

A more unusual version of *dulce de leche* called "Pingo de leite" is found in Brazil. It is a kind of *dulce de leche* with firm outer layer and a soft creamy inside. This texture is obtained by the rapid cooling of the product and partial dehydration while it is spread in thin layers in special trays (STEPHANI *et al*., 2019).

In parallel with the *dulce de leche* paste reported earlier, it is also possible to obtain the *dulce de leche* in bars or tablets. Among the differences in production, the composition of total solids and sucrose content can be noted. For tablets, the amount of sucrose and lactose is higher, which causes an increase in the crystallization process. Also, the product is stirred and cooled at the end of the process, which is necessary to achieve the desired consistency, enhance formation of sugar crystals and generate the desirable characteristics for this kind of *dulce de leche*. The production process is longer than the regular *dulce de leche* paste, leading to an increase in production values due to the higher energy consumption required. However, the use of sucrose increases the product's yield (STEPHANI *et al*., 2019). The maximum permitted moisture for this product is 20 % (w/w) (MERCOSUL, 1996).

The different types of *dulce de leche* found in the legislation result in different types of products and uses. Common *dulce de leche* is usually eaten for breakfast, being been spread on bread, biscuits, or consumed directly (Table 2). *Dulce de leche* for confectionery uses can be employed as filling for wafers, pancakes, cakes, pies and alfajores (a type of confectionery, most common in Argentina and Uruguay). As mentioned above, *dulce de leche* might also be used for ice cream production and it is possible to find several other products

with *dulce de leche* flavor, such as candies and chocolates (ZALAZAR; PEROTTI, 2011; PENCI; MARÍN, 2016; STEPHANI *et al*., 2019).

Table 2 – Utilization of the different types of *dulce de leche* and the major difference between the types.

Types of *dulce de leche*	**Difference**	**Utilization of *dulce de leche***
Common	-	Spread on slices of bread and biscuits, usually at breakfast. It is also eaten directly as a dessert
Mixed with fruits, nuts and/or others	Fruits, nuts and/or others	Usually eaten directly as a dessert
Creamy	Fat from the dairy origin	Consumption directly due to its more incorporated taste
For ice cream manufacture	Colorant	Added to ice cream, gelatos, and other frozen desserts
For the confectionery industry	Stabilizers, thickeners, and/or wetting agents are added	Used in the production of confectionery and bakery products, for example, cakes, pies, and alfajores
Diet	Sugar substitutes, e.g. sorbitol	A product option with fewer calories than common. Can be used similarly to common *dulce de leche*
Tablet	Longer production process and higher sugar content	Sold in large tablets and fractioned into pieces for consumption. Also consumed as a dessert
"Pingo de leite"	Texture (firm cover and soft filling)	A little less common, usually when consumed, is as a dessert

Source: Author (2020)

3.5 CHARACTERISTIC AND FLAVORS OF *DULCE DE LECHE*

The production parameters (time and temperature) and the ingredients (mainly sugars and milk components) are used to influence the final characteristics of the product. During the process, non-enzymatic browning reactions occur, mainly Maillard reaction, providing color and texture to the final product, together with a pleasant taste and aroma (ZALAZAR; PEROTTI, 2011). Among the compounds related to the flavor and aroma derived from the

Maillard reaction, detected by Solid-Phase Microextraction and Gas Chromatography coupled with Mass Spectrometry (SPME-GC-MS) technique, it is possible to mention furans and lactones, 2-methyl furan (chocolate flavor), 2-furan methanol (sulfuric aroma), furfural (sweet/woody aroma) and butyrolactone (creamy/oily aroma) (STAROWICZ; ZIELIŃSKI, 2019). The Maillard Reaction also can form antioxidant products during the various stages of the reaction (YÁÑEZ *et al*., 2018). In addition to the formation of these compounds, the temperature and heating time can lead to the formation of 5-hydroxymethylfurfural (HMF), which appears as an indicator of excessive heat treatment. It has been demonstrated in some studies that HMF can influence biological processes in the organism such as disturbing cell growth, cytotoxic, mutagenic and carcinogenic activity. It is considered a toxic compound to humans and efforts are made so that this compound is not detected in the final product or, if present, are in minimal quantity. The efforts that can be made to avoid the presence of this compound are mainly to control its cause, i.e. excessive heating and acid medium, so the temperature must be controlled during the entire production process and use of sodium bicarbonate, in addition to stirring without pauses and paying attention to the pan walls, avoiding the burning of the product and consequently the generation of unpleasant tastes and formation of HMF (FRANCISQUINI *et al*., 2018; STEPHANI *et al*., 2019).

3.6 PHYSICOCHEMICAL AND MICROBIOLOGICAL PARAMETERS

The physicochemical requirements for *dulce de leche* are defined, with maximum values for moisture and ash content, a minimum value for protein and a permitted fat range, except for *dulce de leche* added with cream, where fat content must be higher than 9 % (w/w) (MERCOSUL, 1996; ZARPELON *et al*., 2016).

The microbiological standard for *dulce de leche* is coagulase-positive staphylococci and yeast and molds counts (MERCOSUL, 1996). The processing parameters of *dulce de leche* (high temperatures for long periods and the presence of sugars) result in a product with low water activity (<0.85) which ensures a longer shelf life (HENTGES *et al*., 2010). Good manufacturing practices also make the product safe, but subsequent fractioning and artisanal production without proper hygiene and health conditions may cause contamination and deterioration of the product (HENTGES *et al*., 2010). Table 3 presents the physicochemical requirements and microbiological standards for *dulce de leche*.

Table 3 – Physicochemical and microbiological requirements for *dulce de leche* according Mercosul legislation.

Requirements		
Microbiological	*Coagulase positive staphylococci counts*	n=5 c=2 m=10 M=100
	Yeast and molds counts	n=5 c=2 m=50 M=100
Physicochemical	Moisture (g/100g)	≤ 30
	Fatty Matter (g/100g)	6 - 9
	Ash (g/100g)	≤ 2
	Protein (g/100g)	≥ 5

Source: Mercosul (1996).
n = number of the samples that must be tested for representative sample
c = Maximum acceptable number of sample units with counts between m and M
m = Limit which separates the acceptable product from the product with acceptable intermediate quality.
M = Counts (CFU/g) values above M are unacceptable.

Hentges *et al*. (2010) showed that the survival of pathogenic bacteria could be possible in *dulce de leche* for up to 30 days, making this product a potential carrier of *Salmonella typhimurium*, *Listeria monocytogenes*, *Escherichia coli* O157: H7 and *Staphylococcus aureus*. Greco *et al*. (2018) observed the behavior of a fungus isolated from *dulce de leche*, *Eurotium chevalieri*, which can grow in products with a water activity of 0.74 at a temperature of 25º C. Some *Eurotium* species are considered deteriorating since they alter the product's organoleptic characteristics and nutritional value, and produces secondary metabolites considered toxic (GRECO *et al*., 2018).

The variability of the process and ingredients used in the production can generate several types of products with different values of protein, ash, fat, and moisture (GAZE *et al*., 2015; YÁÑEZ *et al*., 2018). The variability in the colors of the same product (*dulce de leche*) but of different brands can be noticed in figure 1. Gaze *et al*. (2015) studied several brands of *dulce de leche* and observed significant variability in the values of the following parameters: protein, ash, fat, moisture, and pH. In addition to the variations between the brands, some products presented themselves outside the limits established by legislation for *dulce de leche*, which influences the nutritional composition of the product and these lower values could be related to fraud through ingredient substitution. Several cases of fraud in some foods, such as meat, cheese and milk, are reported in the media. In *dulce de leche*, frauds are also reported, the major one being the addition of starch without the proper labeling, causing losses to the consumer, especially financial, because starch does not cause problems to the organism, but is

added as a way to increase yield, reducing the amount of milk and adding starch, which in addition to devaluing the product, leads to reduction of nutritional values (GAZE *et al*., 2015).

Figure 1 – Color variation between different Brazilian brands of *dulce de leche*.

Source: Author (2020)

3.7 PRODUCTION PROCESSESS OF *DULCE DE LECHE*

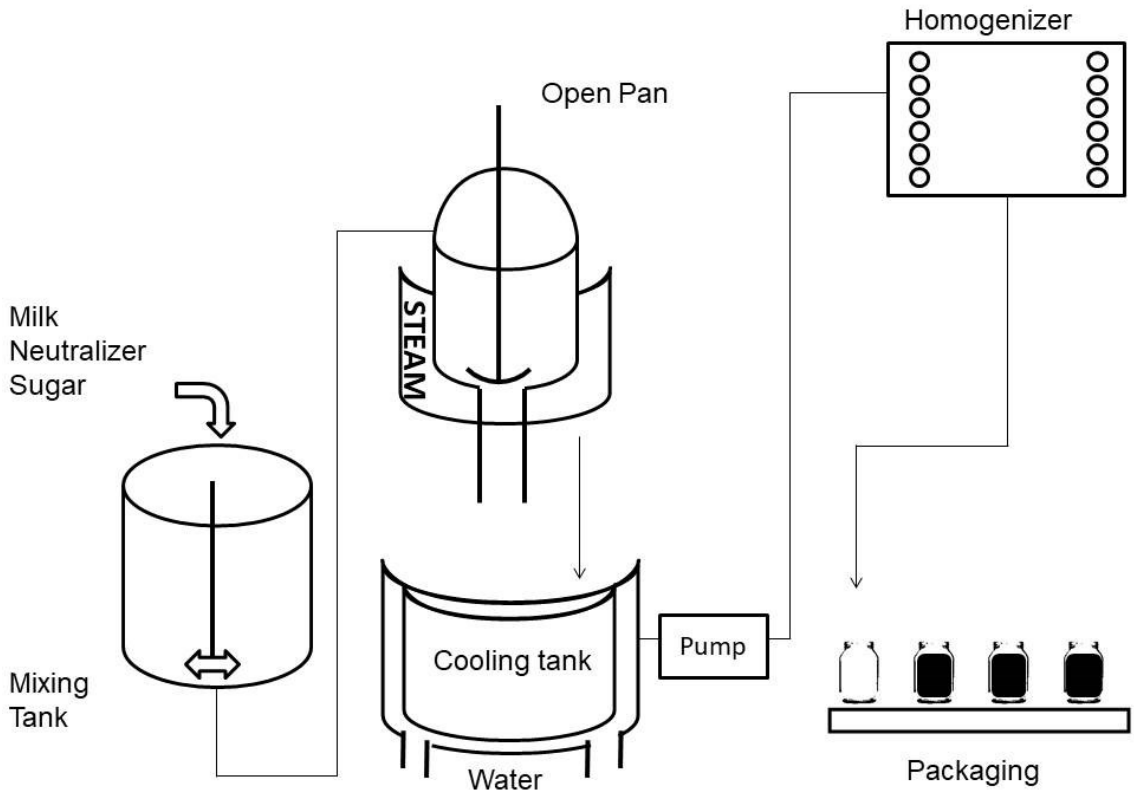
Dulce de leche can be produced in three different ways: traditional open system process, semi-continuous and continuous. The traditional process with the use of a kettle is carried out in batches and is the most widely employed, since, according to producers, the best *dulce de leche* is obtained from this production process, may be for the longer process time and consequently the development of more complex and pleasant flavors and aromas. The first step is the mixing of the ingredients that are preheated to approximately 30 ºC, in order to improve the dispersion of the other ingredients in the milk. The milk can also be previously mixed to alkali agents and then mixed with the other ingredients. A mass balance is performed after deciding the type of *dulce de leche* that will be produced. Ingredients that are used in smaller quantities, such as, alkali agents and flavors are not considered in the mass balance. After finished the mixture, it is gradually heated, to avoid overflowing when boiling, and transferred to the open system, to start the evaporation process (ZALAZAR; PEROTTI, 2011). A closed kettle with reduced pressure can also be used, leading to the evaporation of water under lower heating (40 to 75 ºC) (STEPHANI *et al*., 2019). This part of the process is

where the desirable characteristics of *dulce de leche* are obtained, viscosity, color from non-enzymatic browning reactions (Maillard reaction and caramelization), and flavors. The base of the kettle has a double layer that allows the circulation of steam to heat the mixture. Generally, these kinds of kettles have a capacity of 1,000 L, resulting in 500 kg of *dulce de leche*. The process starts with the addition of only 20 % of the initial mixture, which is heated and concentrated up to 60 % of total solids (measured by a refractometer), and then the remaining mixture is slowly added. The stirrer has an anchor-shaped and heat-resistant plastic paddles, which ensures that the *dulce de leche* does not get stick to the kettle's wall and distributes the heat more effectively.

The concentration process (up to about 60 ºC) is conducted until the total soluble solids reached 68%, as the final concentration (approximately 70%) is reached after cooling. The final concentration can also be determined by a less robust method, where a drop of *dulce de leche* is dripped into a glass of water and its behavior is observed. If the drop dissolves in the water, the process has not finished yet. However, this method is scarcely used and is more common in small productions and by experienced producers. The end of the process is determined when the desired total soluble solids content is reached, and when the mixture has already obtained all the characteristics inherent to the final product in terms of color and sensorial characteristic (ZALAZAR; PEROTTI, 2011; STEPHANI *et al*., 2019).

After concentration, the *dulce de leche* is cooled in a jacketed tank or in the kettle itself, with steam being replaced with cold water. This is an essential step to avoid the most common defect of *dulce de leche*: the formation of crystals that negatively impact its texture. The cooling process starts slowly to support lactose crystallization in beta-anomeric form, with it being the most stable above 93.5 °C. Beta-anomeric is preferred since alpha lactose crystals are larger and more solid than beta lactose crystals. When the temperature falls below 93.5 °C, cooling then occurs quickly to form the smallest possible size of alpha lactose crystals. After cooling, the *dulce de leche* is then sent to a homogenizer to remove any lumps that may have remained, a common occurrence if powdered milk was used, as well as to provide greater brightness and smoothness to the product. The product is packed while still hot (around 50 to 60 °C) in plastic or glass jars (usually for premium products) of different sizes. The products that will be used in the bakery or confectionery industries are generally packaged in larger packs of 10 to 20 kg (HYNES; ZALAZAR, 2009; ZALAZAR; PEROTTI, 2011; PENCI; MARÍN, 2016). Figure 2 presents a scheme of the most common production process for *dulce de leche*.

Figure 2 – Production process of *dulce de leche* in an open system.



Source: Adapted from Zalazar and Perotti (2011).

The addition of ingredients to diversify the types of *dulce de leche*, usually occurs in tandem with the concentration process, so that the added ingredients are also submitted to heat treatment. The most common flavors added to *dulce de leche* are plum, peanuts, coffee, and coconut, among others (MERCOSUL, 1996; STEPHANI *et al*., 2019).

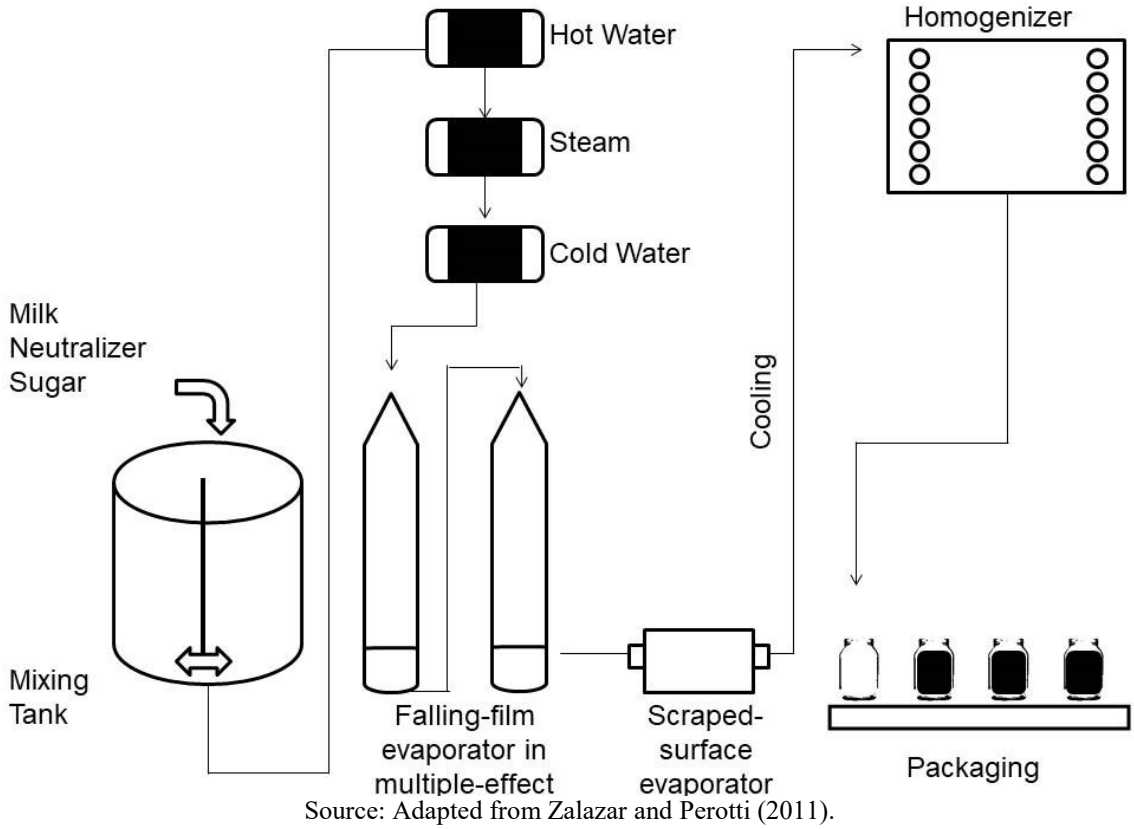
3.7.1 Semi-continuous and continuous production

The semi-continuous process aims to reduce steam consumption and processing time by using a multiple-effect evaporator where the milk and sugar mixture is pre-concentrated. After the multiple-effect evaporator, the concentrate is carried to an open system and follows the process as an open system (batch) (ZALAZAR; PEROTTI, 2011; PENCI; MARÍN, 2016).

The continuous process has two main objectives, to produce *dulce de leche* on a large scale (large volumes) and to reduce steam consumption during the process. However, it is only used in large industrial plants and even so is rarely used since the open system process is considered to produce *dulce de leche* with higher quality. The continuous process occurs oppositely to the batch process and the semi-continuous, starting in the same way, with the

mixture of the ingredients, but product's color is adjusted first and then the concentration itself. The color is obtained through heating the mixture, going through three stages, the first (85°C) and the second heating (110 to 150°C for 1 minute) and the final cooling (50 to 55 ºC). At the end of the heating step, the product already has the desirable color but is still in liquid form, which is taken to a multiple-effect evaporator (e.g. falling-film evaporator), double or triple, and is concentrated to approximately 55 % of total solids. An optional scraped-surface evaporator can be used for concentration up to 70 % of total solids. The final homogenization and packaging process are carried out according to the continuous and semi-continuous process (ZALAZAR; PEROTTI, 2011; PENCI; MARÍN 2016). Figure 3 presents a scheme of the continuous production process of *dulce de leche*.

Figure 3 – Continuous production process of *dulce de leche*.



Source: Adapted from Zalazar and Perotti (2011).

3.8 DEFECTS IN *DULCE DE LECHE*

Among the defects found in *dulce de leche* is acidification, something widely known and common in cheese production. This defect is characterized by the loss of casein stability and subsequent separation into two heterogeneous fractions (protein and whey). Inadequate

pH can also lead to the formation of lactose crystals relatively earlier when compared to the typical crystallization time, and also destabilize Maillard's reaction, where a slightly alkaline pH is considered ideal (MERCOSUL, 1996; HYNES; ZALAZAR, 2009; ZALAZAR; PEROTTI, 2011).

However, the most common defect found in *dulce de leche* is, by far, the crystallization of lactose, which modifies its sensory perception and aids the development of plastic flavors due to the migration of compounds from the packaging to the product. It is also possible to mention the reduction of the nutritional value (loss of lysine) and production of toxic compounds due to the reactions that occur in the environment that can still lead to excessive darkening when the production process does not have proper temperature and time control. *Dulce de leche* with rough texture, syneresis, lack of texture (weak *dulce de leche*) are also reported as defects (HYNES; ZALAZAR, 2009; ZALAZAR; PEROTTI, 2011; PENCI; MARÍN, 2016).

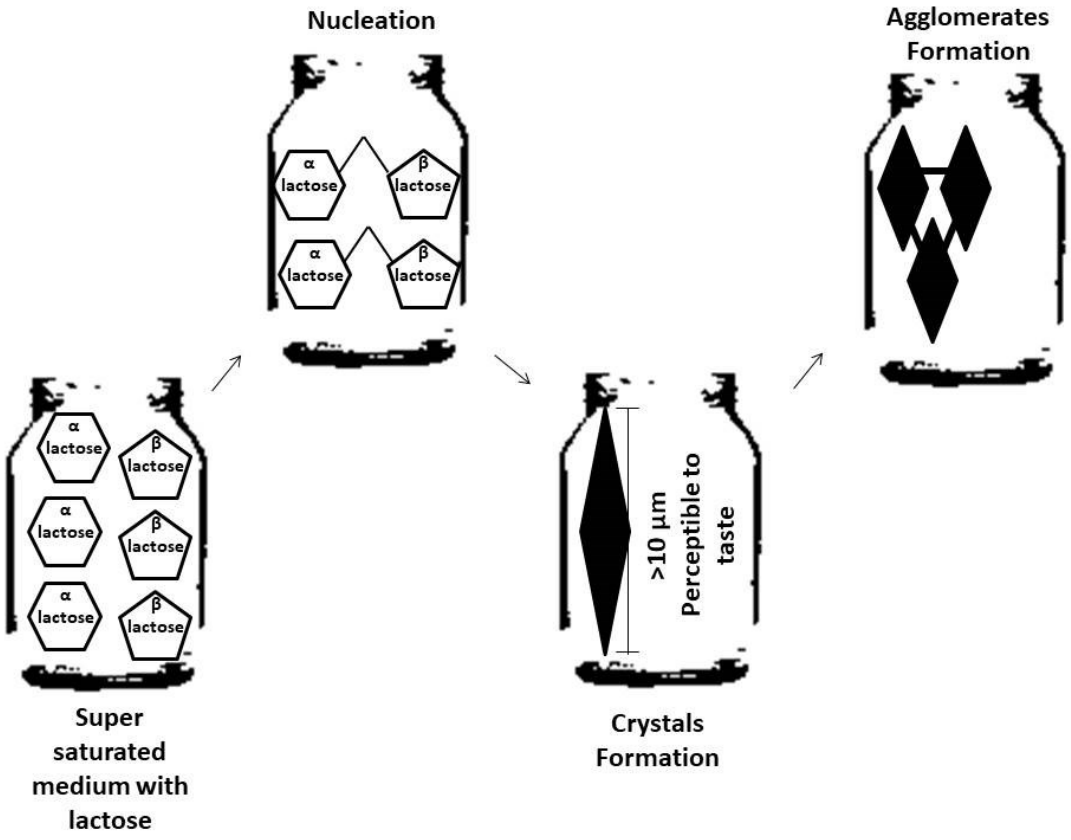
During and after the production of *dulce de leche*, changes that will generate a final product with a desirable characteristic might happen. Some problems could occur during the production process and generate some defects in the final product. It is already known that milk has proteins and sugars (some of them reducing sugars) which, when heated, leads to non-enzymatic browning. This browning is desirable only in *dulce de leche* among all dairy products. Although the Maillard reaction is desirable in *dulce de leche*, it causes a change in nutritional value since the aminoacid lysine found in the medium is reduced (ZALAZAR; PEROTTI, 2011).

Sucrose is not considered a reducing sugar, in contrast to glucose and lactose which reacts with the amino group of proteins, producing a brown color through the Maillard reaction. Caramelization also occurs due to the presence of sucrose. It is worth mentioning that the browning reactions continue during storage, although significantly slower than when under heat. As for the color of the final product, obtained through the non-enzymatic browning reactions, when CIELAB color scale is used for color analysis in *dulce de leche*, the greatest variation is observed in the parameter L, a luminosity parameter that varies from black to White. The variation is observed tending both ways of the parameter, either increasing or decreasing the parameter L (ZALAZAR; PEROTTI, 2011; PENCI; MARÍN 2016). Both reactions are responsible for providing the product's main characteristics of color, aroma, and flavor (HYNES; ZALAZAR, 2009; ZALAZAR; PEROTTI, 2011; PENCI; MARÍN, 2016).

Lactose crystallization, which occurs due to a supersaturated lactose medium, has been previously mentioned and its occurrence can be prevented or reduced by controlling the cooling process. In *dulce de leche* that has undergone the correct cooling process, the formation of crystals larger than 10 μm (greater crystal sizes are perceptible to the consumer) does not occur until 120 days of storage below 20 °C (HYNES; ZALAZAR, 2009; ZALAZAR; PEROTTI, 2011).

The temperature and saturation of the medium are two determining factors for lactose crystallization in *dulce de leche*. Chemically, lactose crystals are formed due to the supersaturation of the medium. A nucleus is formed, as shown in figure 4, and from this, the crystal becomes larger (until it becomes noticeable and characterizes a production defect). The nucleus can grow in two ways: alone or by making connections with nearby nuclei, forming agglomerates that have strong connections between them (ARES; GIMÉNEZ, 2008; HYNES; ZALAZAR, 2009).

Figure 4 – Didactic scheme of the lactose crystallization in *dulce de leche*.



Source: Author (2020)

Considering that the production process already makes the medium supersaturated with lactose, the temperature directly influences the way the crystals are formed. During

storage, crystals continue to be formed and it is important to evaluate the their characteristics: number, growth rate, number and growth rate of crystals agglomerates due to the relationship between product texture, temperature, and the crystals formed (ARES; GIMÉNEZ, 2008; HYNES; ZALAZAR, 2009). Low temperatures (- 5 to 35 ºC) lead to a larger number of crystals, but smaller in size, while the increase in temperature results in fewer and larger agglomerates. The temperature is related to the crystal's formation due to their influence on the texture of the product. As the product texture increases, the motility of the crystals is reduced, thus reducing the formation of the crystals and agglomerates (ARES; GIMÉNEZ, 2008).

To prevent crystallization, lactose should be cleaved into glucose and galactose using an enzymatic process leading to 30-35% reduction in lactose which increases the period without crystallization up to 180 days. The only disadvantage of this technique is its high price due to enzyme's high cost, being recommended only for export products or for restrictive diets (HYNES; ZALAZAR, 2009; ZALAZAR; PEROTTI, 2011). Therefore, the control of *dulce de leche* crystallization by the industry is done controlling the temperature during the process.

Rough texture in *dulce de leche* occurs due to casein coagulation by using milk with high acidity or excessive pH correction with the use of neutralizing agents. Syneresis occurs due to the high acidity or presence of proteolytic bacteria. *Dulce de leche* with a lower viscosity could be the result of excessive stirring during cooling, glucose excess, and incorrect concentration process. *Dulce de leche* with darker coloration than usual is related to an error in the addition of neutralizing agents and/or long heat processing. It is also mentioned that, when packaged in plastic containers, this may cause a the development of a "plastic flavor" in *dulce de leche* and this defect can occur even before the crystallization of lactose during storage (HYNES; ZALAZAR, 2009; ZALAZAR; PEROTTI, 2011).

3.9 INNOVATIONS IN *DULCE DE LECHE* PRODUCTION

Table 4 presents studies obtained through literature review and which will be more comprehensively described below. The search for innovation is linked to the development of new products based on the basic composition of *dulce de leche*. The objective of the search for innovation is to add value and diversify improving the value of raw materials, such as low consumed fruits, the use of whey, or the use of milk from different animal origin by

incorporation in the product and diversifying the product. Besides the diversification of flavors in *dulce de leche*, it is also possible to observe the search to produce healthier products, with reduced sugar content or the changes in the lipid profile.

Table 4 – Studies about different *dulce de leche* and their distinctions when compared with the common.

Dulce de leche	**Country**	**Milk**	**Different ingredientes**	**Collaboration for new products**	**Author**
Low-Fat	Argentina	Skim milk	Xanthan Gum	A reduced-fat *dulce de leche* with the rheological characteristics similar to common	RANALLI; ANDRÉS; CALIFANO (2017b)
No addition of sugar	Brazil	Whole milk	Sucralose, Saccharin, Cyclamate, Sorbitol, Carrageenan	An alternative product targeting consumers who want to reduce the consumption of sugar either for esthetic or health purposes	MILAGRES *et al.* (2010)
Light functional with coffee	Brazil	Skim milk	Sucralose, Polydextrose, Coffe	An alternative to improve the nutritional quality using fiber and sugar reduction	GUIMARÃES *et al.* (2012)
Bubaline *Dulce de leche*	Brazil	Whole buffalo milk	Prebiotic (oligofructose)	A new product to diversify bubaline dairy products.	CARDOSO *et al.* (2018)
Enriched with emulsified pecan oil	Argentina	Skim milk	Xanthan gum, Lactase, Pecan Oil, Tocopherols	An alternative product to common *dulce de leche*, but with a lipid profile of unsaturated fatty acids.	RANALLI; ANDRÉS; CALIFANO (2017a)
***Dulce de leche* from goats' milk**	Brazil	Goats' Milk	Chia Flour	Chia can be a promising thickening agent and resulted in the presence of omega 3 and 6 in the product	CHAVES *et al.* (2018)
***Dulce de leche* from goats' milk**	Brazil	Goats' Milk	-	A product to enhance the use of goats' milk	WANDERLEY *et al.,* (2005)
With Brazilian Savannah fruit araticum	Brazil	-	Araticum pulp, grated coconut	A product for enhancing the value of ingredients consumed by residents and sensorially accepted by children	ARRUDA *et al.* (2016)
With Whey	Brazil	-	Liquid and powder	A product with the use of whey that does	MADRONA *et al.*

			whey	not alter the physicochemical and sensory quality	(2009)
Rinse water treated with nanofiltration and reverse osmosis	Brazil	Whole milk	Recovered milk solids	A viable product for reducing the disposal of dairy industry effluents	BRIÃO *et al.* (2019)
With Whey	Paraguay	-	Whey, Lactase	A product with whey and reduced lactose content with similar characteristics to the common	COHENE *et al.* (2016)
With milk submitted to ohmic heat treatment	Brazil	-	Milk submitted to ohmic heating	A new *dulce de leche* with the application of emerging technology	SILVA *et al.* (2020)

Several relevant studies are focusing on the diversification of *dulce de leche* products, but attention needs to be paid to current legislature, especially if the author is interested in introducing a new product to the market. Studies must comply with the legislation and respect the established identity and quality parameters (MERCOSUL, 1996).

Ranalli, Andrés e Califano (2017b) developed a *dulce de leche* with different moistures (23,7 to 60,3 %) and contents of xanthan gum (0.173 to 0.555%) (as a fat substitute) to observe which combination behaves more similarly to regular *dulce de leche* (without fat reduction). The author concluded that by decreasing the water available in the product, it increased the viscosity and shear flow of the *dulce de leche*. With the proposed methodology it was possible to choose the best combination to obtain a low-fat *dulce de leche* with rheological characteristics like regular *dulce de leche*.

Milagres *et al*. (2010) developed a *dulce de leche* with no addition of sucrose, testing a formulation with sucralose (B), another with cyclamate, saccharin, and sorbitol (C). Regular *dulce de leche* (A) was used as a control to compare the new formulations (B and C) and evaluate the consumer acceptance of a product with reduced sugars. The most globally accepted formulation was A, followed by B and C, these showing similar acceptance but with greater preference for B, which also displayed good acceptance by the tasters for flavor, i.e., developing a healthier *dulce de leche*. It is a novel product, targeting consumers who want to reduce their intake of sugar for aesthetic or health purposes.

Guimarães *et al*. (2012) developed and described a functional *dulce de leche* light added with coffee (1 % instant coffe), to evaluate the effect of dietary fiber concentration and sucralose replacement using physicochemical analysis. A total of 11 formulations with different levels of polydextrose (dietary fiber) and sucralose were developed. The polydextrose, being hygroscopic, increased both the product's moisture and water activity. It was observed that the presence of sucralose (0,1987 to 0,5513) and polydextrose (47,7 to 132,3) does not have a significant influence on the desirable characteristics of *dulce de leche*, but is a novel alternative to improve the nutritional quality of *dulce de leche*.

Cardoso *et al*. (2018) replaced sucrose in a bubaline *dulce de leche* with oligofructose as a prebiotic to observe the influence of this substitution on the physicochemical properties, color, texture, and on the final yield. The bubaline *dulce de leche* with oligofructose did not present a significant difference for acidity, total soluble solids, water activity, mineral salts, and moisture values. An increase in yield and pH was observed with a darker color and a predominance of yellow. A firmer and more adhesive product was

obtained, factors caused by the presence of oligofructose. All these benefits were obtained by replacing 10% sucrose with 10% oligofructose at the beginning of the process. This product brings as an innovation, the valorization of bubaline milk by using it in the production of *dulce de leche*, besides the addition of healthier ingredients to the product.

The authors Ranalli, Andrés e Califano (2017a) produced a *dulce de leche* like product with the addition of pecan oil (23 g for liter) as a source of fat, skim milk, xanthan gum, sugar, and tocopherols to achieve a healthier product. The physicochemical and rheological characteristics remained similar to regular *dulce de leche*, with a slight increase in viscosity only after two months of storage and lipid oxidation after 138 days (stored under room temperature), probably due to the presence of tocopherols and some compounds from the Maillard reaction that may present antioxidant activity, contributing to the product's stability. The lipid profile consisted, mostly, of unsaturated fatty acids (linoleic and oleic), with a reduction in the amount of saturated fatty acids, which was the major reason for the addition of pecan oil. Finally, the study was able to produce a product like *dulce de leche* but with a novel lipid profile characterized by unsaturated fatty acids (RANALLI; ANDRÉS; CALIFANO, 2017a).

Chaves *et al*. (2018) developed a *dulce de leche* made from goat's milk to apply chia flour and partially defatted chia flour in substitution of starch and observe the ideal total solids concentration, as well as evaluate the product by instrumental analysis and sensory analysis. The fat profile, when compared to regular *dulce de leche* was altered, increasing the amount of mono and polyunsaturated fatty acids. The products also showed higher moisture, probably due to water retention by the fibers from chia (1 g / L), as well as due to their difficult solubilization. Color and sensory parameters were significantly different when compared to *dulce de leche* added with starch. The authors infer that chia flour can behave as a novel thickening agent for application in a healthier *dulce de leche*.

Some authors searched for innovations in the variety of products, using new ingredients such as milk from other species that are less common than cows' milk, or incorporating other ingredients such as fruits and, consequently, leading to diversity in products similar to *dulce de leche*.

Wanderley *et al*. (2005) aimed to develop a *dulce de leche* using goats' milk to evaluate the sensory acceptance of the product and the processing time. It was observed that the production process of goats' *dulce de leche* was slightly faster than regular *dulce de leche* made from cow's milk. Goats' milk has a lower moisture content, and its fatty acid

composition has 48 % more saturated short to medium-chain fatty acids when compared to cows' milk, this could justify the shorter process time since the thermal conductivity of saturated fatty acid increases with the reduction of the carbon chain size. Sensory analysis has shown that the most accepted product was formulation B, which contained goats' milk and cows' milk. Formulation C, in addition to having a slightly faster production process than the others, was also well accepted, since it did not differ significantly from B in the acceptance test. Goat's *dulce de leche* can be a new alternative in product diversification and offer a new use for goat's milk as a raw material in the production of dairy products.

Ferreira *et al*. (2020) elaborated an innovative *dulce de leche* made by sheep's milk with and without the addition of sheep's milk cream. These authors stated that the addition of cream to sheep *dulce de leche* did not change the physicochemical and microbiological parameters between formulations, that showed dark color, and sheep *dulce de leche* were stable up to 150 days of storage for most of the evaluated parameters, except for mold and yeast over the evaluated period.

Lima *et al*. (2020) obtained and characterizing "*dulce de leche*" prepared with sheep's and cow's milk in different proportions and noted that the combination of sheep's milk and cow's milk is a technical and nutritionally viable alternative, as it considerably increases the yield of the product. However, sensorial properties and the cost-effectiveness of this product should be considered.

Arruda *et al*. (2016) aimed to develop *dulce de leche* formulations with Savannah fruit (Araticum) and coconut to evaluate the acceptance of these formulations by children (aged 7 to 9 years old). The acceptance by the children varied from likes moderately and like very much. The work proved to be promising because the product was evaluated by the children, a group that generally prefers sweet foods. The innovation aspect of the study focused on adding fruit to the product and thus improving its nutritional quality. It was possible to create a novel product that disseminates fruits from the Brazilian cerrado, which are usually only consumed locally.

Besides the product variability generated by the addition of ingredients that diversify *dulce de leche*, it is also possible to use low-cost ingredients such as whey, turning something that is often treated as effluent into a product used as an ingredient in the production of *dulce de leche*, adding value to the ingredient and the final product as well.

Madrona *et al*. (2009) added whey (powdered and in natura) to *dulce de leche* and aimed to measure the sensory acceptance of the new product. It was concluded that the new

products were well accepted by consumers, and there were no significant differences between the acceptances of *dulce de leche* formulations containing whey, but significant differences in physicochemical properties were observed. Considering the acceptance observed, the authors concluded that it was possible to use whey a substitute for part of the milk, which is an excellent source of protein, without negatively impacting the sensory parameters.

Brião *et al*. (2019) submitted rinse water from the dairy industry to the processes of nanofiltration and reverse osmosis, being possible to recover milk solids and use them in *dulce de leche* and milk-based fermented beverages. The reverse osmosis retentate was pasteurized and added to *dulce de leche* as a partial replacer for milk in 10 %, 20 %, and 30 %. The values for physicochemical parameters remained inside the ranges defined by *dulce de leche*'s legislation in all samples produced with different percentages of retentate, and the final yield of the product was slightly higher with the use of the retentate, showing its application as a viable way to reduce effluent disposal from the dairy industry and add value to rinse water.

Cohene *et al*. (2016) evaluated formulations of *dulce de leche* with a 30 % replacement of milk by whey and evaluated the use of lactase in this formulation. When comparing the physicochemical properties with a standard *dulce de leche*, it was observed that the products that partially replaced milk with whey showed reduced values of fat and protein as well as a lower yield. Concerning sensory analysis, the formulation with whey and enzyme obtained better acceptance by the judging panel, suggesting that *dulce de leche* is a product with potential for the application of certain quantities of whey without affecting the final characteristics of the product.

Several innovations were previously mentioned when it comes to new products and flavors in *dulce de leche*, but the production techniques are quite like the standard production method. It is already known that milk is a matrix rich in several components, which also means that it can be easily modified by the action of microorganisms as well as the interactions between the milk's components themselves (mainly action of enzymes). The processing of milk and its dairy products makes it possible to preserve the product for a longer period as well as to provide different characteristics to the product, sometimes desirable and sometimes not. Despite some techniques having beneficial effects, alterations in the organoleptic characteristics as well as in the nutritional value of the product may occur, also, certain techniques may require careful evaluation in order to determine if the potential benefits outweigh other negative aspects, such as high energetic expenditure, which directly

generates an increase in the final product's price. Due to the deleterious effects caused by thermal processing on dairy products, new techniques are being developed. These emerging technologies seek to improve the production process of dairy products, reducing the negative effects of regular processing techniques by reducing or eliminating the heat treatment stage (AHMAD *et al*., 2019).

Among the emerging technologies, it is possible to mention: application of ultrasound, a simple and affordable technique, which improves homogenization, however, it can generate free radicals that are undesirable in the product. Other application is the high pressure treatments that maintain the nutritional properties of the product are also an option, however, most of the enzymes are very resistant to pressure, and may not be completely inactivated, furthermore, if not performed correctly, high pressure treatments can cause a cascade of reactions that are undesirable in the processing. Application of irradiation/ultraviolet light, although effective in inactivating enzymes and microorganisms, requires high doses of radiation, which can cause negative effects on the chemical and sensory properties of food, as well as being expensive; The application of a pulsed electric field has also become an option for non-thermal treatment, due to its capacity of microbiological and enzymatic inactivation without further damage to the food, but can cause leakage of metals from the electrode to the food, the technique also has a high operation cost, in addition to a series of downsides. Thermosonication deals with the combination of techniques (ultrasound and moderate temperature) in order to increase the effectiveness of the treatment, especially with the use of the techniques mentioned above combined with moderate heat treatment. This treatment has proved to be more effective than the use of a single technique, but it is still worth mentioning the high cost of applying these treatments (AHMAD *et al*., 2019).

Several beneficial effects are observed when using these emerging technologies. One of the reported effects is to improve the food safety of milk, which is a very rich matrix that benefits the growth of pathogenic and deteriorating bacteria. There are already several applications of these technologies directly to milk, but for *dulce de leche* there are few reports of the application of these techniques yet. As previously mentioned, it is extremely important that food is microbiologically safe, in the case of *dulce de leche* for confectionery, after the production process it will still be subjected to a new production process, this time as raw material for another product, so it must have very low microbiological load because it will still be manipulated after its production. Another defect cited previously is the use of heat treatment in the production of *dulce de leche*. It has several beneficial effects such as the

development of desirable flavors and aromas for this product, but on the other hand, there is the production of undesirable compounds that are toxic to the organism, and if there is no complete control of the temperature during the process, the burning of the product can develop unpleasant flavors. Therefore, it is of interest to the industry to develop and apply non-thermal treatment technologies. Among the technologies of application interest, we can mention sonication, irradiation, ultraviolet radiation, pulsed electric field, among others

In general, the previous treatments are commonly applied to milk and some dairy products, and the authors had access to a limited number of studies dealing with the application of emerging technologies in *dulce de leche* production, being a scientific approach to be followed and discovered in a way that could generate further studies and advances in *dulce de leche* production, making this product more accessible and standardizable.

Silva *et al*. (2020a) assumed that milk for *dulce de leche* production is heat-treated to reduce the microbiological load of the raw material and the temperature causes injuries in the dairy matrix. Thus, the author sought to apply the emerging technology of ohmic heat treatment and to evaluate the sensory characteristics of this new *dulce de leche* with the use of advanced sensory tools. The authors were able to conclude that the sensory analysis tools were able to characterize *dulce de leche* as well as show the changes of attributes over time. Silva *et al*. (2020b) affirmed that ohmic heating can preserve food quality while inactivating pathogens and spore-forming microorganisms. The ohmic treatment with a slight to moderate electric field did not influence the product's acceptability, but there was a change in flavor, giving the product a pronounced bitter taste and reducing in the characteristic aroma of *dulce de leche*. By using a high-intensity electric field, there was an increase in the sensory characteristics, as well as an increase in the product's brightness, fluidity, and flavor, reduced consistency and texture, and an overall increase in acceptance by the consumers. In general, the sensory perceptions were more accentuated as the strength of the ohmic treatment increased.

Considering the compilation of the above-mentioned studies, it should be noted that part of the studies involving *dulce de leche* focus on new additives, flavors, and different raw materials. The emerging technologies offer potential to improve the production process of *dulce de leche* since they have several advantages when compared to common heat treatments, as well as their application combined with the other stages of the process can improve efficiency and reduce costs. Studies are still needed to demonstrate how these emerging technologies influence *dulce de leche* (AHMAD *et al*., 2019).

The area of food research and development is in constant evolution due to the need and demand of consumers for new products and innovations, either by the simple desire for different products or by the need to adapt diets. The existence of diseases such as gluten allergy, lactose intolerance, diabetes, or the search for healthier habits drives the industry to develop more and more different products. The increasing number of lactose-free or reduced-sugar product options observed in the markets shows that in addition to new products, new production processes have emerged to develop these products with quality for the consumer, developing new production technologies. Besides these, there is the interest in the development and application of emerging technologies that already have some studies, showing beneficial effects, but it is still necessary to expand the range of application and evaluate, in addition to the beneficial effects, if there is any deleterious effect on the product, which is only a negative effect or the development of toxic compounds.

4 MATERIAL E MÉTODOS

O doce de leite se apresentou como um produto relativamente escasso de evidências e estudos científicos quando comparado à demais derivados lácteos como iogurtes, bebidas lácteas, queijos, dentre outros. Está afirmação pode ser confirmada com uma busca rápida ao site *Science Direct* utilizando a palavra "*Dulce de leche*", recuperando um total de 189 documentos, quando alteramos a busca para "cheese" o site recupera 93.261 documentos. Realizando a mesma busca anterior no site *Springer,* recuperamos 66 documentos referentes a doce de leite e 56.261 referentes a queijos. Desta forma, foi necessário fazer um apanhado geral e elaborar métodos de busca que recuperassem a maior quantidade possível de dados e artigos para serem utilizados como informações durante a escrita do artigo. Foram escolhidas diversas palavras referentes ao doce de leite as quais foram utilizadas como busca em diversos bancos de dados científicos. As palavras utilizadas na busca para recuperação de dados e artigos do doce de leite foram "*Dulce de leche*", "Doce de leite", "*Milk jam*", "*Arequipe*" e "Manjar blanco" buscando assim maior quantidade de dados em diversas línguas. Além da busca de cada palavra, foi feito também a junção de duas ou mais palavras de buscas através da utilização de parênteses, colchetes e as conexões e/ou. Os bancos de dados utilizados para busca de informações foram: Science Direct, Springer, Wiley, Scielo, Scopus, Portal Capes, Taylor e Francis e Google Acadêmico. O período de busca estabelecido foi para artigos mais recentes ou a partir do ano de 2008, sendo utilizado artigos que apresentassem assunto referente ao assunto, uma vez que mesmo com a utilização das palavras específicas, artigos não inerentes ao assunto foram recuperados.

Com este apanhado geral na metodologia de busca, foi possível resgatar uma quantidade significativa de dados que embasasse a escrita deste artigo, tornado este uma fonte de dados confiável para consulta de informações sobre o doce de leite, buscando a disseminação de informações e dados científicos sobre este produto de sabor único e ainda tão pouco difundido.

5 RESULTADOS E DISCUSSÃO

Foi possível observar que mesmo com a utilização de diversas palavras referentes ao doce de leite e a busca destas em variados banco de dados, observou-se uma relativa escassez de dados quando comparados a produtos lácteos como queijos, iogurtes, bebidas lácteas e outros. Este dado pode estar relacionado com a baixa disseminação a nível mundial de consumidores deste produto quando comparado novamente com os produtos citados anteriormente.

Conforme foi observado na revisão bibliográfico, o doce de leite é difundido e consumido principalmente em alguns países da América do sul, com breve disseminação em poucos países da Europa, já os queijos, por exemplo, possuem disseminação mundial. Além deste fato, podemos considerar que o doce de leite possui um processo produtivo mais robusto e "simples" quando comparado aos queijos, por exemplo, que possuem diferentes formas de produção, diferentes fermentações, diferentes tempos de maturação. Também é possível citar as bebidas lácteas que possuem diversos sabores e texturas, logo estes produtos podem despertar o interesse maior da indústria devido aos diferentes potenciais destes produtos, levando o doce de leite a ocupar posições inferiores no interesse de industrialização e consequentemente compra e venda.

Mesmo com os fatos citados anteriormente, é necessário chamar atenção para o doce de leite, pois seus sabores e aromas são inigualáveis, além de, conforme já citado anteriormente, possuir processo produtivo relativamente simples, tornando possível de ser produzido em indústria de pequeno à grande porte, além de possuir fácil adaptação, sendo possível alterar uma linha produtiva para que a mesma comece a ser utilizada para produção do doce de leite.

A legislação vigente para este produto é completa e indica todos os padrões de identidade e qualidade para o doce de leite, porém a legislação se encontra desatualizada quando considerado seu ano de emissão sendo 1996, sendo interessante o desenvolvimento de uma comissão para reavaliar e promover alterações e atualizações na legislação. Os processos produtivos também já se apresentam bem estabelecidos e determinados para cada produto específico, juntamente com os adjuvantes de processo para cada tipo específico de produto.

As inovações tem se mostrado como uma boa opção para inovações de produtos e processos produtivos, o que também poderia ser levada em consideração no desenvolvimento e elaborações de uma nova legislação mais atual e inclusiva para produtos inovadores.

6 CONCLUSÃO

É necessária a disseminação de informações sobre o doce de leite, pois é relativamente fácil de produzir quando comparado a outros produtos lácteos, tem um sabor adocicado, agradável e é fácil de ser comercializado devido a sua longa vida útil e por não necessitar de refrigeração. A produção do doce de leite envolve o aquecimento e concentração do leite e do açúcar, desenvolvendo características, em sua maioria causadas pela reação Maillard, que transformam o doce de leite em um produto único e saboroso. O processo de produção e os principais ingredientes já estão bem estabelecidos, reduzindo o risco de defeitos e erros no processo produtivo. Além disso, o doce de leite tem diversas aplicações tanto em produtos de confeitaria quanto para consumo direto. Vários estudos já demonstraram que o doce de leite é também uma matriz potencial para inovação. Consequentemente, o aumento da diversidade de produtos pode levar à valorização de matérias-primas pouco disseminadas e outros ingredientes pouco utilizados em produtos lácteos. Finalmente, espera-se que este trabalho possa informar o leitor e auxiliar na disseminação deste produto latino-americano, bem como apontar formas de evitar defeitos durante a produção e armazenamento do doce de leite.

7 SUGESTÃO PARA TRABALHOS FUTUROS

A autora sugere como trabalho futuro, uma nova busca de dados referentes ao doce de leite e observar quais são as potenciais inovações e estudos deste produto que ainda não foram realizadas. A autora sugere como formas de inovação, considerando o destaque da utilização de probióticos e a sua importância na saúde humana, a realização da aplicação destes no doce de leite. Considerando seus empecilhos tecnológicos (temperatura), este produto se comportaria não mais como uma matriz de probióticos, mas sim como uma matriz para paraprobióticos. Logo, o doce de leite é um potencial produto para avaliar como o probiótico se comportará frente ao aquecimento e quais os potenciais benefícios destes quando se tornam paraprobióticos, se os benefícios seriam similares aos ofertados pelo consumo de probióticos. Aplicação das técnicas moleculares para avaliação da taxa de sobrevivência bem como para avaliação da presença do DNA dos probióticos, buscando comprovar que mesmo que a bactéria viável não esteja mais presente, potenciais benefícios poderiam ser obtidos da ingestão deste produto, logo a autora sugere também análises ligadas a saúde, avaliando como o consumo dos paraprobióticos pode beneficiar o organismo humano.

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ANEXO A – *Dulce de leche*: product types, production processes, quality aspects, and innovations

Neste anexo está exposto o artigo: *Dulce de leche: product types, production processes, quality aspects, and innovations*, o qual foi aceito para publicação em dezembro de 2020 na revista *International Journal of Dairy Technology* – IJDT (Qualis Capes para CIÊNCIA DE ALIMENTOS – B1).