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Effect of the unit operations: evaporation and drying on the whole and skimmed milk powder properties

Sara Carolina Gallegos Bosmediano
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SARA CAROLINA GALLEGOS BOSMEDIANO

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Dissertation submitted to the Chemistry Engineering Graduate Program of the Universidade Federal de Viçosa in partial fulfillment of the requirements for the degree of *Magister Scientiae*.

Adviser: Rita de C. S. de Sousa

Co-adviser: Kely de Paula Correa

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Dedicated to the women of my family—my ancestors, my grandmothers, my mother,
and my sister. Your unconditional love will always live in my work.

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ABSTRACT

BOSMEDIANO, Sara Carolina Gallegos, M.Sc., Universidade Federal de Viçosa, January, 2025. **Effect of the unit operations: evaporation and drying on the whole and skimmed milk powder properties.** Adviser: Rita de Cassia Superbi de Sousa. Co-adviser: Kely de Paula Correa.

Milk is a nutrient-dense drink with high protein, bioavailable amino acids, vitamins, and essential minerals like calcium. The demand for powdered milk has increased due to its lack of refrigeration and extended shelf life, making it a cost-effective preservation method. Concentration and drying are crucial in milk powder production, influencing milk constituents such as fat, protein, and lactose. These may undergo structural and chemical changes that impact physical and functional properties, including flowability, compressibility, packaging, stability, reconstitution, color, browning index, and foaming. This study analyzes the effects of milk type, evaporation equipment, and drying techniques on milk powder properties. A full factorial design (2³) was used, with factors at two and three levels. The milk types tested were whole milk and skim milk. Evaporation was performed with an open-pan evaporator and a rotary evaporator, at 100.33°C and 60°C. Drying was done using a spray dryer at 150°C and a freeze dryer at -45°C. Both the rotary evaporator and freeze dryer used vacuum systems, reducing the boiling point during concentration and aiding water sublimation in low-pressure environments. A total of eight treatments were produced. The study analyzed the chemical composition of liquid milk and powder, as well as microstructure using Scanning Electron Microscopy (SEM). Additionally, bulk and compact density, Hausner ratio, Carr's index, water activity, hygroscopicity, solubility, wettability, color parameters, Browning Index, and foaming properties were assessed. ANOVA and Tukey's test were used for statistical analysis. Results showed that milk type significantly affected flowability, density, stability, color, and foaming properties. Skim milk powder, with higher protein and lower fat content, had greater compactness and a higher Carr Index (CI), associated with poor flowability. It also absorbed more moisture due to higher protein and lactose levels, with smaller particles that enhanced solubility and wettability. Water activity and hygroscopicity were influenced by amorphous lactose and protein. Foam stability increased with higher protein content, while fat in whole milk powder reduced foaming. Evaporation and drying methods also affected powder density. The open-pan evaporator and spray drying produced denser particles with better flowability, but increased moisture content, which could reduce stability and promote browning. The high processing temperatures raised

the Browning Index (BI), causing a color shift towards red and yellow tones due to Maillard reactions. In contrast, the rotary evaporator and freeze dryer, with lower temperatures and vacuum systems, minimized thermal damage, preserving the color and stability of the powder. Freeze-drying resulted in more porous and hygroscopic powders. These findings provide valuable insights for optimizing milk powder production, helping the dairy industry balance cost, efficiency, and quality.

Keywords: spray dryer; freeze dryer; packing; flowability; stability; foaming

RESUMO

BOSMEDIANO, Sara Carolina Gallegos, M.Sc., Universidade Federal de Viçosa, janeiro de 2025. **Efeito das operações unitárias: evaporação e secagem nas propriedades de leite em pó integral e desnatado.** Orientadora: Rita de Cassia Superbi de Sousa. Coorientadora: Kely de Paula Correa.

O leite é um alimento nutritivo, rico em proteínas, aminoácidos biodisponíveis, vitaminas e minerais essenciais, como o cálcio. A demanda por leite em pó cresceu devido à sua longa vida útil e à dispensa de refrigeração, tornando-se um método econômico para sua conservação. A concentração e secagem influenciam a distribuição e as interações de componentes como gordura, proteína e lactose, podendo alterar suas propriedades físicas e funcionais, como fluidez, compressibilidade, estabilidade, solubilidade, umectação, cor, índice de escurecimento e capacidade de formação de espuma. Este estudo investigou os efeitos do tipo de leite, equipamento de evaporação e técnicas de secagem nas propriedades do leite em pó. Utilizou-se um delineamento fatorial completo (2^3), com três fatores em dois e três níveis. Os dois tipos de leite foram integral e desnatado. A concentração foi realizada com evaporador de panela aberta ($100,33^{\circ}\text{C}$) e evaporador rotativo (60°C). A secagem foi feita em spray dryer (150°C) e liofilizador (-45°C). O uso de vácuo nos dois sistemas de concentração e secagem reduziu o ponto de ebulição e facilitou a sublimação da água. Foram realizados oito tratamentos e analisados a composição química do leite líquido e do leite em pó, além da microestrutura, por Microscopia Eletrônica de Varredura (MEV). A densidade aparente e compactada, razão de Hausner, índice de Carr, atividade de água, higroscopicidade, solubilidade, umectação, parâmetros de cor (L, a, b*), índice de escurecimento e propriedades de formação de espuma também foram avaliados. A análise dos fatores foi realizada por ANOVA e teste de Tukey. Os resultados mostraram que o tipo de leite influenciou a fluidez, densidade, estabilidade, cor e propriedades de espuma. O leite em pó desnatado, com mais proteína e menos gordura, teve maior compactidade e índice de Carr (CI) mais alto, associado a baixa fluidez. Além disso, absorveu mais umidade devido ao maior teor de proteína e lactose e apresentou partículas menores, melhorando a solubilidade e umectação. A atividade de água e a higroscopicidade foram influenciadas pelos níveis de lactose e proteína. A estabilidade da espuma aumentou com maior teor de proteína, enquanto o alto teor de gordura no leite integral reduziu a formação de espuma. Os métodos de evaporação e secagem também influenciaram a densidade do pó. O evaporador de tacho aberto e o spray dryer produziram partículas mais densas e com

melhor fluidez, mas aumentaram a umidade residual, o que pode reduzir a estabilidade e promover o escurecimento. As altas temperaturas aumentaram o Índice de Escurecimento (BI), mudando a cor para tons avermelhados e amarelados devido à reação de Maillard. Em contrapartida, o evaporador rotativo e o liofilizador, com temperaturas mais baixas e sistemas de vácuo, minimizaram danos térmicos, preservando melhor a cor e a estabilidade. A liofilização resultou em pós mais porosos e higroscópicos. Esses resultados oferecem insights valiosos para otimizar a produção de leite em pó, equilibrando custo, eficiência e qualidade.

Palavras-chave: spray dryer; liofilizador; embalagem; fluidez; estabilidade; formação de espuma

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LIST OF ABBREVIATIONS AND ACRONYMS

| | |
|------------|--|
| CI | Carr's Index |
| HR | Hauser Ratio |
| BI | Browning Index |
| L* | Lightness or darkness of the sample. |
| a* | Parameters represent the red-green axis. |
| b* | Parameters represent the yellow-blue axis. |
| Aw | Water activity |
| SEM | Scanning Electron Microscope |
| WOS | Whole milk powder is produced using an open-pan evaporator and spray dryer. |
| WRS | Whole milk powder is produced using a rotary evaporator and spray dryer. |
| WOF | Whole milk powder is produced using an open-pan evaporator and freeze dryer. |
| WRF | Whole milk powder is produced using a rotary evaporator and freeze dryer. |
| SOS | Skimmed milk powder is produced using an open-pan evaporator and spray dryer. |
| SRS | Skimmed e milk powder is produced using a rotary evaporator and spray dryer. |
| SOF | Skimmed milk powder is produced using an open-pan evaporator and freeze dryer. |
| SRF | Skimmed milk powder is produced using a rotary evaporator and freeze dryer. |

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1. INTRODUCTION

Milk is regarded as a highly complete food, which plays a relevant role in the diet of over 7 billion people (GÓRSKA-WARSEWICZ et al., 2019; HEMME, 2022). According to SMITH et al. (2022), milk offers a significant amount of high-quality protein, bioavailable amino acids, vitamins, and essential minerals like calcium. Additionally, milk is a primary contributor to global nutrient availability, providing 49% of calcium, 24% of vitamin B2, 18% of lysine, and 15% of dietary fat (SMITH et al., 2022).

Brazil ranks as the third-largest milk producer globally, following only India and the USA (BARROS et al., 2022). The country ranks fifth in global cow milk consumption and second in the powdered milk market, with consumption rising in recent years, reaching approximately 621 thousand tons by July 2022 (ROSA; PRUDENCIO, 2023). The increased demand for powdered milk is due to lack of refrigeration needs, significantly extended shelf life, and lower transportation costs compared to liquid milk. As a result, the production process for powdered milk has become one of the most common and cost-effective methods for preserving milk. (DE OLIVEIRA et al., 2021).

Concentration and drying are essential to produce milk powder. Concentration involves evaporation, that occurs at the product's boiling point (FARKYE, 2019). During this process, the substance is heated, causing water to change into a gaseous state, which is then carried away. In contrast, drying is commonly defined as the extraction of water through mass and heat transfer from a food product. This process involves the movement of liquid to the surface, where it evaporates and is removed (WOO; BHANDARI, 2023). These processes, both concentration and drying, can affect the structure and composition of milk components, causing variations in their properties (KERR, 2019).

Evaporation can lead to whey protein denaturation, altering air occlusion and particle density (LIN et al., 2018). Drying causes liquid milk to convert into solid particles with different surfaces, which consists of amorphous lactose, fat globules, casein micelles, and whey proteins. These transformations can alter the powder's chemical structure and physical properties such as bulk and compact density, flowability, cohesiveness, water activity, hygroscopicity, color, and foaming capacity and stability, which are critical for storage, handling, packaging, and reconstitution. (ZOUARI et al., 2020).

Bulk and compact density directly influence packing properties, lowering storage and transportation expenses (DING et al., 2020). Flowability and cohesiveness affect the

powder's behavior during handling, storage, and processing (FOURNAISE et al., 2020). Water activity and hygroscopicity are crucial parameters for maintaining milk powder quality, as they regulate microbial growth and chemical reactions while influencing moisture absorption, which impacts storage stability and drying (RYABOVA; SEMIPYATNY; GALSTYAN, 2023).

Reconstitution properties like solubility and wettability are essential for the powder to dissolve, which affects the texture and usability of the final (WANG et al., 2016). The color of milk powder is another important factor, as it can influence consumer purchasing decisions and serve as an indicator of the product's chemical composition, freshness, and processing efficiency (BOJANA MILOVANOVIC et al., 2020). Foaming capacity refers to the powder's ability to form foam when mixed with water, while foaming stability determines how long the foam maintains its structure. Both properties are influenced by protein content, which stabilizes foam structure (MEENA et al., 2017).

Extensive research has focused on the effects of drying on milk powder characteristics, particularly spray drying, the primary technique, used in the dairy industry for the production of milk powder. However, studies have also explored the effects of freeze drying on milk powder properties, even though this method is mainly employed to produce high-value powdered compounds, such as lactoferrin. For instance, DESHWAL et al. (2020) investigated the *"Effect of spray and freeze drying on physico-chemical, functional, moisture sorption, and morphological characteristics of camel milk powder,"* and BALDELLI et al. (2022) explored the *"Spray freeze drying of dairy products: Effect of formulation on dispersibility."* However, these studies mainly focus on drying methods, with limited attention to the combined effects of milk type, concentration, and drying on milk powder properties. This reveals a gap in understanding how these factors influence milk powder's physical, chemical, and functional characteristics.

Given the importance of milk powder production and consumption and the need for insights into how these processes affect quality, production, processing, storage, and transportation., this work aims to bridge this gap. It evaluates the effects of milk type (whole and skimmed) and unit operations—concentration (open pan and rotary evaporator) and drying (spray and freeze drying)—on the chemical composition, physical properties, functional characteristics, and morphological traits of milk powder.

GENERAL AND SPECIFIC OBJECTIVES

2.1 General Objective

Assessment of the effect of unit operations—evaporation and drying—on the chemical composition, physical, functional, and morphological characteristics of whole and skimmed milk powder.

2.2 Specific Objectives

- Analysis of the Chemical Composition of Liquid Milk and Powdered Milk Samples
 - Characterization of the Chemical composition of Whole and Skim milk
 - Preparation of Whole and Skim milk for powder production
 - Characterization of the Chemical composition of Whole and Skim milk powder
- Evaluation of the Microstructure of Whole and Skim Milk Powder
- Assessment of the Physical and Functional Properties of Whole and Skim Milk Powder
 - Analysis of flow, compressibility, and packaging properties: bulk density, tapped density, Carr index (CI), and Hausner ratio (HR)
 - Evaluation of the Stability and reconstitution properties: water activity, hygroscopicity, solubility, and wettability
 - Analysis of color properties: color parameters and browning index.
 - Assessment of foaming properties of Whole and Skim milk powder.

3. LITERATURE REVIEW

3.1 Milk and dairy products

Milk stands as one of the most versatile ingredients in the food industry. It is made up of about 87% water, 4-5% lactose, 3% protein, 3-4% fat, 0.8% minerals, and 0.1% vitamins (GÓRSKA-WARSEWICZ et al., 2019; SMITH et al., 2022). Moreover, milk is a great source of high-quality protein, vitamins, and minerals and a relevant source of essential amino acids essential for human nutrition (BEZIE, 2019).

ERCAN; TEL ADIGÜZEL (2022) mentioned that milk is an essential food for bone health because of its protein and calcium compounds. Not drinking cow's milk or consuming it in low amounts during childhood has been associated with a higher risk of bone fractures before puberty. Drinking cow's milk regularly during childhood is linked to greater bone density in adulthood according to ERCAN; TEL ADIGÜZEL (2022).

Studies suggest that consuming milk and dairy can help prevent periodontal disease, reduce cholesterol absorption, and manage body weight and blood (LEE; KIM, 2019). Research has also linked low-fat milk with lower risks of hypertension, dental caries, colon cancer, heart disease, and stroke (BABIO et al., 2015). As a result, the market for skimmed products has grown in recent decades (HAYES et al., 2023). Despite this, whole milk's richer sensory quality compared to skim milk contributes to its continued popularity (SCHIANO; HARWOOD; DRAKE, 2017).

Nowadays, powdered products are widely chosen because they do not require refrigeration and have a longer shelf life (TASTEMIROVA et al., 2020; DESHWAL et al., 2020). Dehydrated dairy products represent an innovative approach with more convenient and practical application (ROSA; PRUDENCIO, 2023). Moreover, drying is one of the most widely used and cost-effective methods for preserving milk, allowing it to be transported easily and safely at a lower cost than liquid milk. (DE OLIVEIRA et al., 2021).

The consumption of powder products is increasing according to the United States Department of Agriculture (ROSA; PRUDENCIO, 2023). Dairy powders serve multiple purposes, whether reconstituted or used as ingredients in various food products, such as meat, bakery, and confectionery items (ROSA; PRUDENCIO, 2023). Concentration and drying are used to produce powdered products. Concentration removes water from milk, often through evaporation, before drying. Drying then converts the concentrated milk into powder, typically through spray drying (KERR, 2019).

3.2 Evaporation

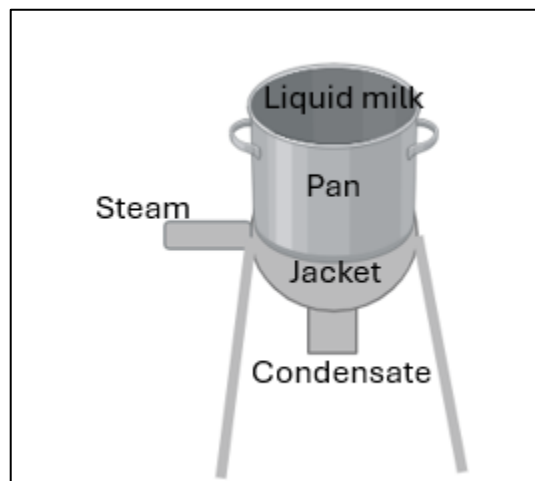
Concentration involves removing water from milk, typically through evaporation, to increase the solids content to approximately 40–50%. It is often employed as a preliminary step before drying, offering a more cost-effective method compared to spray drying (LIN et al., 2018). Several factors must be considered during this process, including the reduction of nutritional values, steam and water requirements of the equipment, and process control and monitoring (FARKYE, 2019).

Evaporators can be categorized based on various criteria, including the heater, heating medium, and the flow of milk. Small-scale evaporators, like rotary evaporators, save energy and water by operating efficiently at lower temperatures (MAURÍCIO et al., 2021), while batch and pan-type evaporators more straightforward and less expensive, they have limited use in the dairy industry due to factors such as higher energy consumption, labor intensity, and space

(ROY et al., 2017). In contrast, large-scale industrial units predominantly use falling film and multiple-effect evaporators (FARKYE, 2019), which are favored in the dairy industry for their high heat transfer coefficients, energy efficiency, and flexible operation. However, they can be expensive and complex in design, consuming relatively high energy (ROY et al., 2017).

An open-pan evaporator is a simple, low-cost method for concentrating on milk. It consists of a chamber where milk is placed, surrounded by a jacket filled with saturated steam to provide heat for evaporation. As water is removed, the milk solids become more concentrated. The vapor produced during this process rises directly from the chamber into the surrounding air (KERR, 2019), as seen in Figure 1.

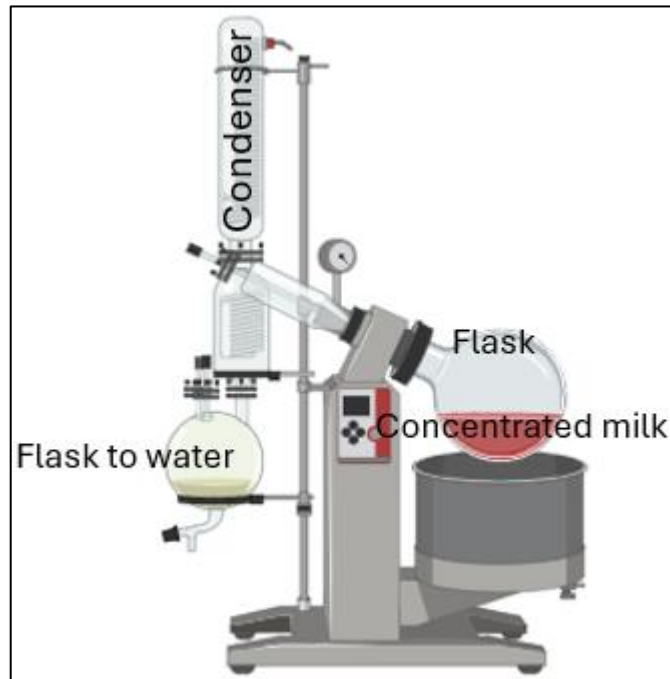
Figure 1-Evaporation system using an open pan evaporator



Adapted from: KHOLER, (1955)

Rotary evaporators, on the other hand, are efficient alternatives for concentrating milk. Water evaporates as the milk rotates in a flask due to heat from a water bath. The temperature is lower than in an open pan evaporator because the vacuum system reduces the pressure inside the equipment. The evaporated water is then directed to a condenser, condensing back into a liquid and collecting in a separate flask (MAURÍCIO et al., 2021), as seen in Figure 2, which shows the components of a rotary evaporator

Figure 2-Evaporation system using a rotary evaporator.



Adapted from: HAMBBA et al. (2014)

The boiling temperature of milk depends on its total solids (TS) content, including proteins, fats, lactose, and minerals. An increase in TS results in a higher boiling point, a phenomenon known as boiling point elevation, which is a colligative property. The dissolved particles hinder the escape of water molecules into vapor, requiring more energy for the liquid to reach its boiling temperature. This phenomenon is described by the following equation, which represents the boiling point rise of a solution, where \times denotes the total solids (TS) content (Geankoplis, 2003).

$$T_{boiling} = 100 C^{\circ} + 1,78 \times + 6,22 \times^2$$

3.3 Techniques of drying

Drying is the process of removing water from a food product through heat and mass transfer. Spray drying is commonly used in the dairy industry to dry concentrated milk, with hot air facilitating the removal of water. This process continues until the milk reaches approximately 95% total solids, transforming it into powder (WOO; BHANDARI, 2023). Spray drying is an efficient and cost-effective method (SCHUCK et al., 2016; ZOUARI et al., 2020b). However, it can degrade heat-sensitive nutritional components (GÓRSKA-WARSEWICZ et al., 2019).

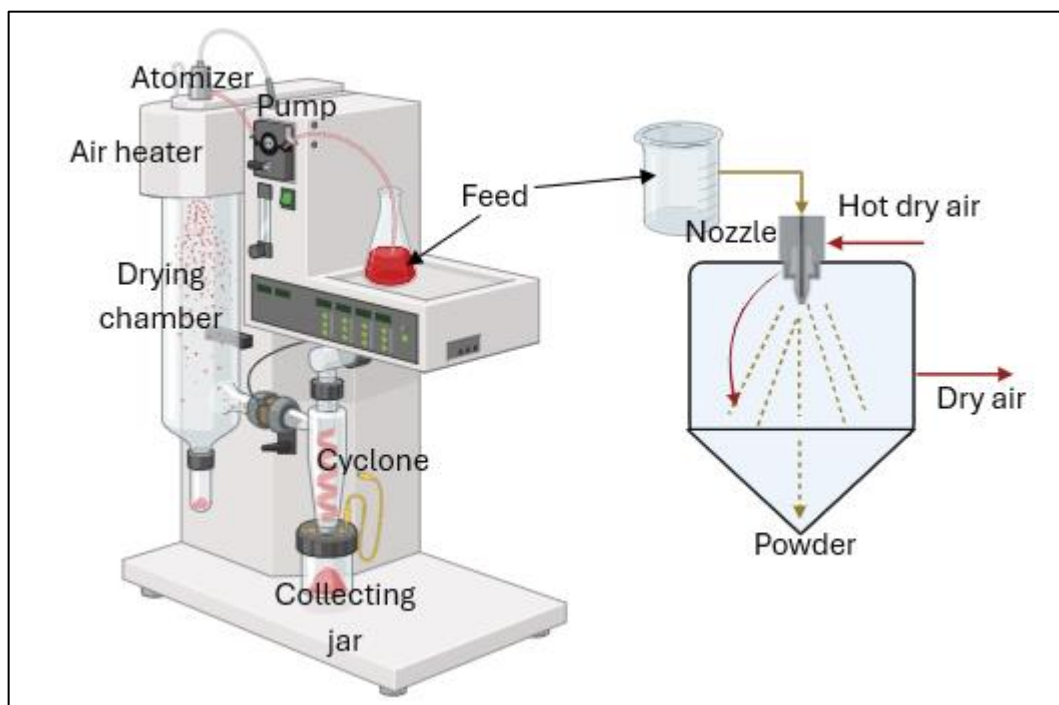
In contrast, freeze drying is often used to process high-value bioactive compounds in milk, such as lactoferrin. This technique not only enhances the shelf life of these products but also

preserves their sensitive biofunctional properties (WANG et al., 2017). By operating at low temperatures, freeze drying preserves heat-sensitive compounds and prevents non-enzymatic reactions, such as protein denaturation and Maillard reactions. However, DESHWAL et al. (2020) highlighted that freeze drying is a costly operation.

3.3.1 Spray drying

Spray drying is a unit operation used to produce milk powder from concentrated milk in a cost-effective and time-efficient manner. During this process, the feed flows from the product feed tank to the atomizing device at the top of the drying chamber. There, it is atomized into fine droplets and mixed with flow of hot, dry air, leading to rapid evaporation of moisture. As drying progresses, the moisture content decreases, causing the solution to reach saturation. The dissolved solids then solidify and form a solid layer on the droplet's surface, ultimately leading to the formation of milk powder (WOO; BHANDARI, 2023), as seen in Figure 3.

Figure 3-The dryer system that uses a spray dryer.



Adapted from: OAKLEY (2004)

After drying, within the chamber most of the dried product falls to the bottom and moves into a pneumatic conveying and cooling system. The powder is then separated in a cyclone before being bagged for storage or distribution (WOO; BHANDARI, 2023). After the spray drying, fluidized beds can be used to remove residual moisture until a moisture content specific or cool the powder to prevent degradation and to prepare it for packaging and finally this equipment can agglomerate fine particles into larger, more flowable, and easily rehydrated

granules (SIVAKUMAR et al., 2016).

During spray drying, the balance between Dry-Bulb Temperature (DBT) and Wet-Bulb Temperature (WBT) is critical for ensuring efficient drying and maintaining milk powder quality. A higher DBT accelerates water evaporation, while a lower WBT indicates drier air, which enhances moisture removal (MAHMOUD, 2014).

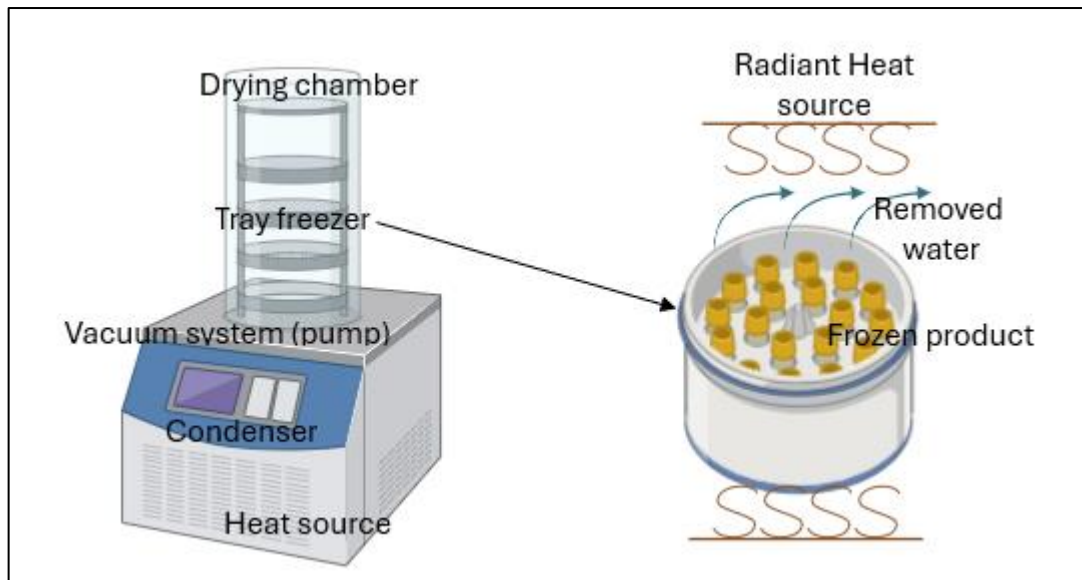
The spray drying conditions could exert significant influence over the properties of the resulting powder. Types of dryers, atomization pressure, feed concentration, feed flow, and inlet air temperature play key roles in determining the characteristics of spray-dried milk powder (SAHA; NANDA; YADAV, 2019).

3.3.2 Freeze drying

Lyophilization, commonly known as freeze drying, is a process conducted at low temperatures. The freeze-drying consists of three primary stages: freezing, sublimation, and desorption, which are carried out in freeze-dryers. Four key components make up the design: a drying chamber, a vacuum pump, a heat source, and a condenser (GARCIA-AMEZQUITA et al., 2015). First, Sublimation occurs concurrently with freezing at temperatures around -50°C in the drying chamber, while the vacuum pump removes non-condensable gases from the chamber. Once the frozen product is under high vacuum, the heat source provides the latent heat necessary for sublimation (GAIDHANI et al., 2021).

Once all the ice has sublimed, the secondary drying phase begins, during which water is desorbed from the remaining cake. The heat used in this phase is conductive or radiative heat, applied gradually to increase the product temperature while maintaining vacuum conditions (GAIDHANI et al., 2021). Like most of the ice sublimates, bound water continues to be removed from the product through desorption. Finally, the milk powder is collected, while the condenser captures the water released from the ice in the product (GARCIA-AMEZQUITA et al., 2015) as seen in Figure 4.

Figure 4-The dryer system that uses a freeze dryer.



Adapted from: SAJAL (2012)

Because freeze-drying occurs at low temperatures, it can preserve heat-sensitive compounds and prevent whey protein denaturation and the Maillard reaction. The resulting product from freeze drying has high shelf-stability due to its low moisture content and rigid, porous structure (DESHWAL et al., 2020). Research by IBRAHIM; KHALIFA (2015) demonstrated that the nutritional properties of the powdered product remain unchanged compared to fresh milk, demonstrating the efficacy of freeze-drying for producing high-value products such as coffee, microorganisms, encapsulated aroma (UEDA et al., 2023).

3.4 Characteristics and Properties of Dairy Powders

The usefulness of milk powder depends on both their physical and functional properties, including powder structure and particle size distribution, flowability, compressibility, packing, stability, reconstitution, color and functional properties. These characteristics significantly impact transportation, handling, processing, and powder formulation. Both concentration and drying are used to produce powdered products. These processes facilitate the movement of milk constituents, notably fat, protein, and lactose, toward the surface of the particles, which can significantly affect their physical and functional properties (KELLY; O'CONNELL; FOX, 2016).

According ANEMA (2019) the concentration by evaporation could influence the level of whey protein denaturation in milk. Undenatured whey proteins increase air occlusion, while denatured proteins result in higher particle density. On the other hand, WOO; BHANDARI (2023) mentioned that during the drying, droplets of liquid milk can transform into solid

particles with distinct surfaces, which can affect the milk powder properties. Thus, understanding the chemical structure changes during concentration and drying is essential for assessing their impact on manufacturing, applications, nutritional attributes, key chemical differences, and functional properties (ANEMA, 2019; WOO; BHANDARI, 2023).

3.4.1 Chemical and morphological changes during drying

During drying, milk constituents, particularly fat, protein, and lactose, migrate toward the particle surfaces influencing their physical and functional properties (WOO; BHANDARI, 2023). Changes in the chemical structure of milk components are closely related to properties such as particle size distribution, reconstitution (wettability, solubility), foaming, hygroscopicity, water activity, powder density, bulk density, particle density, and flowability (KELLY; O'CONNELL; FOX, 2016).

ANEMA (2019) noted that spray drying can cause denaturation of whey proteins, exposing hydrophobic regions that may lead to aggregation and reduced solubility in water, which can affect their foaming ability (DESHWAL et al., 2020). FARKYE (2019) stated that heat exposure during spray drying influences fat oxidation, which subsequently impacts the shelf life of the powder.

Lactose in dairy powders often exists in an amorphous state can lead to plasticization, when it interacted with water contributing to powder stickiness and caking. Manufacturers can enhance milk powder quality by controlling drying conditions, such as water content and temperature, to prevent the plasticization of amorphous lactose (CARPIN et al., 2016). Additionally, the lactose can react with free amino groups in proteins, peptides, or free amino acids through the Maillard reaction (HUPPERTZ; GAZI, 2016) .

3.4.2 Microstructure of Milk Powder

The microstructure of milk powder can be analyzed using Scanning Electron Microscopy (SEM), which produces high-resolution images of the surface. This technique provides detailed information about particle size, morphology, surface texture, and agglomeration (BURGAIN et al., 2017). The milk powder particle size significantly influences its appearance, reconstitution properties, compressibility, packing characteristics, and flow behavior (DESHWAL et al., 2020; ZAFISAH et al., 2018).

3.4.3 Flowability, Compressibility, and Packing properties

Bulk Density and Compact (tapped) Density

Several properties influence the flowability, compressibility, and packing characteristics of milk powder, with density being a relevant factor. Density, defined as the mass of the powder per unit volume, can be categorized into two types: bulk density and compact (tapped) density (PUGLIESE et al., 2017). Higher density typically indicates better packing of particles, which is influenced by their size, shape, distribution, and how well they fit together. Increasing bulk density can improve packing efficiency, making it more economical to transport and store, as less volume is required for the same mass of product (ZOUARI et al., 2020c).

Hausner Ratio and Carr's Index

The flowability and cohesiveness of milk powder are commonly assessed using the Hausner Ratio and Carr's Index, which refer to how easily the powder moves under gravity or other forces and cohesiveness states to how much the powder particles stick together, forming lumps instead of flowing freely. High cohesiveness can lead to poor flowability, causing blockages in equipment and difficulties in storage, handling, and processing (FOURNAISE et al., 2020). According to SUHAG; KELLIL; RAZEM (2024), bulk density, compact density, and their ratios (e.g. Hausner Ratio and Carr's Index) are widely used to measure these properties. The following Table 1 shows the Hausner Ratio (HR) and Carr Index (CI) values along with their corresponding flowability classifications in milk powder.

Table 1. Flowability Classification Based on HR & CI

| Flowability | Hausner Ratio (HR) | Carr Index (CI, %) |
|----------------|--------------------|--------------------|
| Excellent | 1.00 – 1.11 | 0 – 10 |
| Good | 1.12 – 1.18 | 11 – 15 |
| Fair | 1.19 – 1.25 | 16 – 20 |
| Passable | 1.26 – 1.34 | 21 – 25 |
| Poor | 1.35 – 1.45 | 26 – 31 |
| Very Poor | 1.46 – 1.59 | 32 – 37 |
| Extremely Poor | ≥ 1.60 | ≥ 38 |

(SEO, 2022)

3.4.4 Stability and Reconstitution Properties

Stability Properties: Water activity and Hygroscopicity

Water activity (WA) refers to the available free water in a product for microbial growth or chemical reactions. In milk powder, a_w typically ranges from 0.11 to 0.23, helping maintain its quality and properties (RYABOVA; SEMIPYATNY; GALSTYAN, 2023). Hygroscopicity refers to the powder's ability to absorb moisture from the environment (JUAREZ-ENRIQUEZ et al., 2017).

The critical factors that influence higher water activity (WA) and hygroscopicity in milk powder are its composition and particle size and structure. The amounts of protein, lactose, and fat play a key role in these properties (SUHAG; KELLIL; RAZEM, 2024). For instance, lactose is the main factor influencing these properties, because it easily absorbs and retains water. It also plays a key role in the glass transition temperature (T_g). Below T_g , lactose remains in a stable, glassy state, limiting moisture movement. Above T_g , it becomes sticky and absorbs more water, increasing WA (HUPPERTZ; GAZI, 2016).

The hydrophilic nature of proteins allows them to bind water, with whey proteins being more water-attractive than casein. Meanwhile, fat acts as a moisture barrier, reducing hygroscopicity and lowering WA. In addition to composition, particle size and structure affect moisture absorption. Finer or more porous particles have a larger surface area, making them more prone to absorbing water from the environment (KELLY; O'CONNELL; FOX, 2016).

Reconstitution Properties: Solubility and Wettability

The ability of the powder to fully dissolve in water is referred to as solubility, which can form a uniform solution, while wettability refers to the powder's capacity to absorb water and become hydrated (KELLY; O'CONNELL; FOX, 2016). Low solubility can leave undissolved particles, causing incomplete rehydration, while poor wettability leads to clumping and uneven mixing. Fat dispersion in milk powder also affects reconstitution, as fat's hydrophobic nature can slow down wetting (FOURNAISE et al., 2020).

3.4.5 Color Properties

CIELAB Color System

Color is an important quality trait in milk powder, affecting consumer choices and indicating its composition, freshness, and processing conditions (BOJANA MILOVANOVIC et al.,

2020). The color of milk powder is measured using the CIELAB color system (L^* , a^* , b^*), which describes how we see color:

- L^* (Lightness): Measures how light or dark the milk powder is, from 0 (black) to 100 (white). For instance, a low L^* value (darker color) may indicate browning due to overheating or Maillard reactions (SULIEMAN et al., 2015).
- a^* (Red Green): Shows the balance between red ($+a^*$) and green ($-a^*$). Negative a^* values may suggest product degradation or contamination (DESHWAL et al., 2020).
- b^* (Yellow Blue): Represents the shift between yellow ($+b^*$) and blue ($-b^*$). A higher b^* value can indicate higher fat content. (DESHWAL et al., 2020).

Browning Index (BI)

The Browning Index (BI) is a value used to measure the browning in milk powder, mainly due to chemical reactions like the Maillard or caramelization. When proteins (amino acids) and reduced sugars react under heat, it causes a non-enzymatic reaction that changes the color (browning), flavor, and nutritional content of the milk powder (AL-HILPHY et al., 2022).

3.4.6 Foaming capacity

Foaming capacity refers to the capacity of milk powder to create foam when mixed with water and agitated, while foaming stability describes how well the foam retains its structure over time (MEENA et al., 2017). The foam capacity and foam stability of milk powder depend on several factors, such as protein content, fat content, air, and water (RYAN; O'REGAN; FITZGERALD, 2023).

Proteins (especially whey proteins) are the main contributors to foam formation because they trap air when the milk powder is rehydrated and mixed. However, if the proteins are denatured, it can result in poor foam stability or clumping. On the other hand, lower-fat milk powder tends to produce better foam capacity because it allows air to be incorporated more easily (HO et al., 2024).

Introducing air into the mixture of milk powder and water helps improve both foam capacity and stability. Additionally, a higher water content can lead to better foam formation, but if there is too much water, the foam may become less stable. The pH of the water (e.g., too acidic or too alkaline) and the processing conditions (e.g., higher temperatures) can also affect protein denaturation, which in turn influences the foam properties (HO et al., 2024; RYAN; O'REGAN; FITZGERALD, 2023).

4. METHODOLOGY

This research was conducted in the fields of Process and Material Development. The methodology was carried out at the Cândido Tostes Dairy Institute, in the Operations and Process Laboratory (LOP) at the Department of Food Technology (DTA), in the Technological Process Laboratory (PROTEC) at the Department of Chemical Engineering (DEQ), and in the SEM Laboratory at the Department of Physics (DPF). The last three laboratories are part of the Universidade Federal de Viçosa (UFV). Additionally, all reagents used were of analytical grade. The whole and skimmed milk were donated by the Instituto de Laticínios Cândido Tostes - EPAMIG. The milk samples were standardized, transported in a cold chain, and stored in a freezer at -20 °C.

Standardization of milk

The standardization process starts with collecting raw milk, which comes directly from cows and naturally varies in fat and protein content. Next, the milk goes through skimming, where a centrifuge separates the fat to achieve the desired fat content—around 3% for whole milk and 0.1–0.5% for skim milk. After that, the milk is homogenized by forcing it through small nozzles at high pressure to evenly distribute fat molecules, preventing separation. Finally, it is pasteurized by heating it to a specific temperature to eliminate harmful bacteria while preserving its nutritional value (KALIT; KALIT; DOLEN[~], 2021).

4.1 Analysis of Chemical Composition of Liquid Milk and Milk Powder Samples

4.1.1 Chemical composition of whole and skimmed milk

The chemical analysis followed AOAC methods (2020). Protein content was analyzed using the Kjeldahl method; fat by the Gerber method; Moisture by gravimetric method in the oven drying at 110°C; ash by gravimetric method in the “mufla” at 550°C; and carbohydrates were calculated by difference.

Protein quantification

Protein content was determined using the AOAC Official Method 991.20. The sample mass was determined using an analytical balance (Dalmaq, Shimadzu AY-220, Minas Gerais, Brazil), and 0.25 grams were transferred to the Kjeldahl tube. A catalytic mixture and concentrated sulfuric acid were added, and the tube was placed on a heating block for digestion, with the temperature gradually increasing from 50°C to 370°C. After digestion, the sample was neutralized with approximately 20 mL of concentrated sodium hydroxide solution (40-50%),

and the distillate was trapped in 20 mL of 4% boric acid solution with four drops of a mixed indicator (methyl red and bromocresol green). Standardized 0.1 N hydrochloric acid was used to titrate this solution.

$$\% p = ((V - V_b) \cdot N \cdot f)_{\text{HCl}} \cdot 14 \cdot \frac{0,6385}{P_s} \quad (1)$$

Where %p is the percentage of protein, V is the volume of HCl (mL), V_b is the blank sample, N is the normality of HCl (N), f is the correction factor for HCl normality, and P_s is the sample mass (g).

Fat quantification

This procedure followed the AOAC Official Method 989.05, which is based on the digestion and extraction of lipids from samples and the measurement using a butyrometer for milk with 0-8% fat content. A weighed 1 g sample was combined with 10 mL of sulfuric acid solution (90%) with a density of 1.5 g/mL. The mixture underwent heating in a water bath (Solab, SL-153, São Paulo, Brazil) until a wine color appeared. The solution resulting from digestion was transferred to the butyrometer and washed with 9 mL of sulfuric acid (90%). Then, the butyrometer was sealed with an appropriate stopper after adding 1 mL of iso-amyl alcohol. After shaking, the samples and reagents were centrifuged at 1200 rpm for 15 minutes in a Gerber centrifuge (KCEN, ITR-SÚPER II, Porto Alegre, Brazil). To obtain the result, the butyrometer was placed in a water bath at 65 °C, and the column reading was adjusted. The value obtained on the scale corresponds directly to the percentage of fat, which should be read at the lower meniscus.

Moisture Quantification

Moisture analysis was performed using the AOAC Official Method 927.05 which involves removing water from the sample by evaporation in an oven (Marconi MA-032, São Paulo, Brazil). A 2 g sample was weighed with an analytical balance, dried at 105 °C for three hours, and subsequently cooled in a desiccator (Lumilabor, São Paulo, Brazil), and their masses were recorded. After another 30 minutes in the oven, the plates were cooled in a desiccator and weighed. The plates remained in the oven for another 30 minutes before being cooled in a desiccator and weighed. Moisture content was determined using the following equation (2), where "PA" represents the initial mass of the sample and plate in grams, and "PAs" represents the mass of the sample and plate after reaching a constant weight.

$$\text{Moisture (\% m/m)} = \frac{(PA - PAs)}{PA} * 100 \quad (2)$$

Ash Quantification

The ash quantification used the AOAC Official Method 945.46. Initially, the porcelain crucible was placed in a muffle furnace (Marconi, MA 385/3, São Paulo, Brazil) at 550 °C for 30 minutes, then cooled in a desiccator and weighed on an analytical balance (m1). A 5 g sample (m0). was weighed into a crucible and placed in a muffle furnace at 550 °C for 4 hours or until white ash formed. Once the ash was confirmed to be white, the crucible was cooled in a desiccator and its mass was measured again (m2) (AOAC,2020). The percentage of ash in the sample was calculated using Equation 4.

$$\text{Ash (\%)} = \frac{(m2-m1)}{m0} * 100 \quad (4)$$

Carbohydrate Quantification

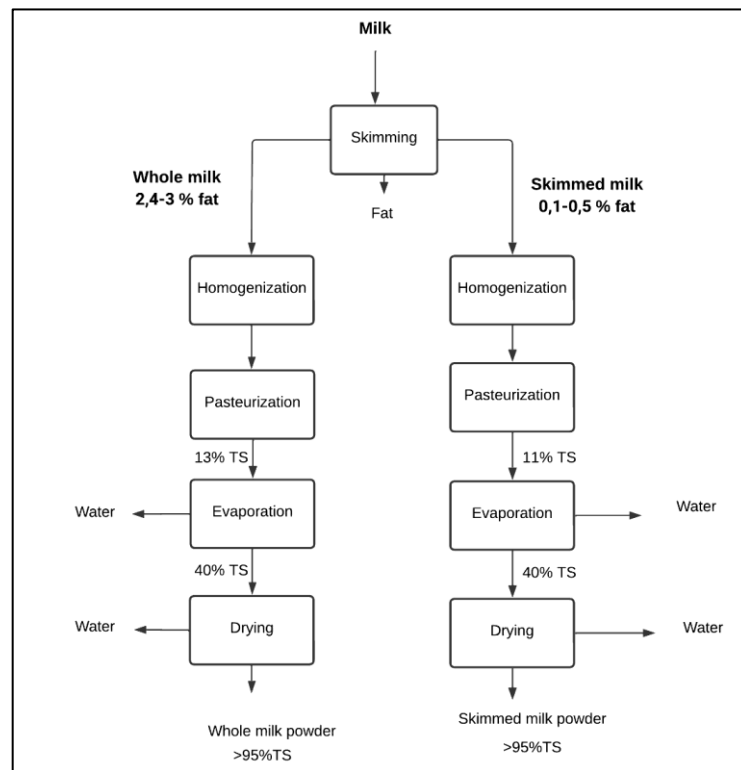
The carbohydrate values in the samples were quantified by calculating the difference in the percentage of the other constituents analyzed, as described in Equation 5

$$\text{Total carbohydrate (\%)} = 100 - \text{Protein} - \text{Fat} - \text{Ash} - \text{Moisture} \quad (5)$$

4.2 Preparation of whole and skimmed milk powder

Both whole and skimmed milk are collected after the standardization process, which includes collection, skimming, homogenization, and pasteurization. Next, the milk powder preparation involved two stages: evaporation and drying, as seen in Figure 5.

Figure 5 - The block diagram for whole and skimmed milk production.





Source: the author

The first stage was concentration by evaporation. The concentration used two types of evaporators: an open pan evaporator and a rotary evaporator and was completed when the total solid content of the milk reached 40%. Total solids (TS) were assessed using a Brix refractometer, which measures sugar concentration to estimate the TS content in milk.

In the open-pan evaporator, the milk was heated to 100.33°C to reach boiling, which is the typical condition for evaporating a solution with 11-13% total solids (TS) at 1 atm, as seen in Table 2. The steam passing through the evaporator jacket was at 157°C and 5.7 atm, providing enough heat to evaporate water from the milk (ROY; BHUSHANI; ANANDHARAMAKRISHNAN, 2017).

The rotary evaporator operated with a manometric pressure of 600 mmHg, which corresponds to an absolute pressure of 0.21 bar ($P_{abs} = 760 - 600 = 160 \text{ mmHg} = 0.21 \text{ bar}$). The water bath reached 74°C, providing enough heat to evaporate water from the milk at 60°C, as seen in Table 2. In a rotary evaporator, by reducing the boiling point of water, the vacuum system enables evaporation at a temperature lower than that of milk's boiling point (MAURÍCIO et al., 2021).

Table 2 -Operating conditions for A) open pan and B) rotary evaporator.

| Equipment | Operation conditions |
|---|---|
| A)  | T_s: 157 °C T_s: steam temperature P_v: 5,7 atm P_v: steam pressure |
| (Mecamau, C-058, Sao Paulo, Brazil) | |
| B)  | T_{sy}: 74 °C T_{sy}: system temperature P_v: 600 mmHg P_v: vacuum pressure of system vt: 70 rpm vt: work velocity |
| (Tecnal, TE 211, Sao Paulo, Brazil) | |



The second stage, drying, used spray drying and freeze-drying techniques under fixed conditions, as seen in Figure 5. This unit operation aimed to obtain powder with 95% total solids, thus reducing the water concentration almost completely. Total solids (TS) were assessed using a refractometer.

The spray drying was carried out at an air temperature of 150°C, as shown in Table 3. During the process, the relative moisture of the inlet air ranged from 10% to 30% and its temperature reached 150 °C, which promoted efficient and fast drying. As a result, the atomized milk droplets quickly lost water and reached a final temperature of around 70°C to 90°C.

The freeze dryer works by combining low temperature (T_{ch}) and low vacuum pressure (P_v), which allow sublimation. The low temperature keeps the product frozen, while the low

vacuum pressure helps the water vapor escape, as seen in Table 3. After processing, the milk powder contained approximately 95% total solids (TS) and was stored at -20°C.

Table 3-Operating conditions for A) spray dryer and B) freeze dryer.

| Equipment | Operation conditions |
|--|--|
| <p>A)</p>  <p>(Pilotech, MSD 1.0, Shanghai, China)</p> | <p>Fp: 0,7 L/h Fp: Feed pump</p> <p>Tinlet: 150 °C Tinlet: air inlet temperature</p> <p>Bc: 0,64m³/min Bc: Blower control</p> <p>% solids: 40% % solids: feed solids percentage</p> |
| Equipment | Operation conditions |
| <p>B)</p>  <p>(JJ Científica, liofilizador-JJ02, São Carlos, Brasil)</p> | <p>Tch: -48 °C Tch: drying chamber temperature</p> <p>Pv: 0,045mbar Pv: vacuum pressure</p> <p>tv: 24 to 48 hours tv: freeze drying tim</p> |

4.3 Chemical composition of whole and skimmed milk powder.

The chemical analysis followed the AOAC methods (2000). Protein content was determined using AOAC Official Method 991.20, moisture content was measured using AOAC Official Method 927.05, ash quantification was done according to AOAC Official Method 945.46, and carbohydrates were determined using different methods. The methodology for measuring the chemical composition is explained in section 2.1.1. However, for milk powder, the Gerber method (AOAC Official Method 989.05) was slightly modified. A tenfold dilution of 10 grams of powder was prepared using distilled water, and 10.75 mL of the resulting

solution was placed in a butyrometer. The procedure then followed the methodology described in section 4.4.

4.4 Microstructure Analysis of Milk Powder

A thin gold coating was applied to the milk powder samples. A conductive adhesive, such as carbon tape, was used to secure the powder onto the stub. The sample was then placed in a sputter coater (Metallizer, Q150RS, Judges House, United Kingdom), where a gold coating (approximately 5-10 nanometers) was applied. This thin coating allowed electrons to escape more easily, improving image quality.

A Scanning Electron Microscope (SEM) was used to examine the microstructure of the milk powder (JEOL, JSM-6010LA, Boston, USA). The sample was placed in the SEM chamber and analyzed under high vacuum conditions. Observations were made at 1000× magnification with a 10-micrometer scale. The average particle diameter was measured using ImageJ software.

For the spray-dried samples, the Analyze Particles function was used to determine the particle diameter based on the average area of circular particles. ImageJ assumes that particles are circular and apply specific conditions for threshold, segmentation, and size. Particles smaller than 1 μm^2 were excluded. Following the area determination, the diameter was obtained using the equation below:

$$D = 2 \times \frac{A}{\pi} \quad (\text{Eq 6})$$

For measuring the diameter of freeze-dried samples, the Freehand Line tool was used to trace the longest dimension of irregular shapes. The program requires certain conditions: the particles must be well-defined, the longest dimension should be traced accurately (avoiding unnecessary curves), and consistency is important as measurements depend on manual accuracy. A total of one hundred measurements were taken, and the average diameter was then calculated.

4.5 Flowability, Compressibility, and Packing Properties Analysis

Bulk density

The method described by JAFARI; GHALEGI GHALENOEI; DEHNAD (2017) was followed to measure bulk density. A 10 mL graduated cylinder was filled with 2 g of powder

without applying any external pressure. The bulk density was then calculated by dividing the powder's mass by the volume of the loosely packed powder.

Compact density

Following the method described by JAFARI; GHALEGI GHALENOEI; DEHNAD (2017), compact density was assessed. In the same way as the bulk density method, 2 g of milk powder was placed in a graduated cylinder. The cylinder was then vibrated for 1 minute using a magnetic stirrer (BS-2H, Labscience, São Paulo, Brazil) to help the particles settle and fill the empty spaces. To obtain the compact density, the powder's mass was divided by its settled volume.

Carr's Index (CI) and Hausner Ratio (HR)

Flowability and cohesiveness were analyzed according to the method proposed by PUGLIESE et al. (2017). Flowability refers to how easily a powder flows, while cohesiveness describes the tendency of particles to stick together. Higher cohesiveness results in poorer flowability.

In this context, the compressibility of the powder is evaluated using Carr's Index (CI) and the Hausner Ratio (HR). Lower CI and HR values indicate lower cohesiveness and better flowability. These indices are calculated based on bulk and tapped density using the following formulas:

$$CI = \frac{\text{Compact density} - \text{Bulk density}}{\text{Compact density}} \times 100 \quad (\text{Eq. 7})$$

$$HR = \frac{\text{Compact density}}{\text{Bulk density}} \quad (\text{Eq.8})$$

4.4 Stability and Reconstitution Properties Analysis

Water activity

At 25 °C, the water activity (WA) of the dairy powders was assessed with an AquaLab CX-2 meter (Apeldoorn, Netherlands). Water activity is a key indicator of how water affects microbial growth and chemical reactions in milk powder. Measuring WA is essential for predicting and controlling the stability and safety of during the shelf life of dairy powders (BALDELLI et al., 2022).

Hygroscopicity

Hygroscopicity was measured according to the methodology outlined by SAMSU; ZAHIRAH; ZAHIR (2020). Two grams of powder were placed in a desiccator (Lumilabor, São Paulo, Brazil) which used a saturated sodium chloride (NaCl) solution to regulate the relative humidity at 75% and the temperature at ~25 °C. The samples were exposed to these conditions for 7 days to allow moisture adsorption. The mass of each sample was taken and recorded before and after the equilibration period to determine the moisture absorbed.

The calculation of hygroscopicity was based on the mass of adsorbed moisture per 100 g of dry solids (sample). This parameter is crucial for evaluating the powder's capacity to absorb moisture from the environment, which directly influences both storage stability and quality.

Solubility

The gravimetric method outlined by JAFARI; GHALEGI GHALENOEI; DEHNAD (2017) was employed to determine solubility. This technique was chosen to minimize potential instrumental errors and eliminate the need for complex calibration procedures.

Initially, 2.5 g of milk powder was combined with 30 mL of distilled water at room temperature in a pre-weighed centrifuge tube. The solution was agitated for 1 minute with a vortex mixer at medium intensity. Next, the sample was placed into a water bath for heating (Solab, SL-153, São Paulo, Brazil) at 37 °C for 30 minutes. It was then centrifuged (Eppendorf, Centrifuge 5430R, Hamburg, Germany) at 877 g (3500 rpm) and 4 °C for 20 minutes.

The supernatant liquid was transferred with care into a pre-weighed dish and dried at 105 °C until constant weight was achieved, meaning the weight variation was less than 0.001 g. Using the following formula, solubility was calculated:

$$\text{Solubility (\%)} = \frac{(\text{Dried supernatant weight})}{(\text{Initial sample weight})} \times 100 \quad (\text{Eq.9})$$

Wettability

The wetting time was employed to assess the hydration characteristics of the milk powder, following the method described by SEO (2022). This method was chosen for its simplicity, reproducibility, and direct measurement. Wetting time was measured as the time it took for 5 g of powder to completely wet and immerse in 100 mL of distilled water at 23 ± 1 °C.

This technique measures the duration it takes for a specified amount of powder to become fully wetted and submerged in water at a controlled temperature. By employing this method, we can effectively evaluate the hydration properties of the milk powder, which is crucial for understanding its rehydration behavior in various applications.

4.5 Color Properties Analysis: Color Parameters and Browning Index

Color Parameters

A colorimeter (Konica Minolta CR-5, Tokyo, Japan) was used to measure the color of eight milk powder samples. This device uses $d:8^\circ$ sphere geometry, which means it applies diffuse illumination and measures the reflected light at an 8° viewing angle. The Konica Minolta CR-5 is compatible with various standard illuminants, including D65, which simulates natural daylight conditions for more accurate color evaluation (MILOVANOVIĆ et al., 2020).

DESHWAL et al. (2020) mentioned that the CIE L^* , a^* , b^* color properties can be reported such as:

- L^* : Lightness (ranges from 0 = black to 100 = white).
- a^* : Red-green axis (Redness is indicated by positive values, and greenness by negative values).
- b^* : Yellow-blue axis (Yellowness is represented by positive values, and blueness by negative values).

Analyzing the color provides essential details about the quality and appearance of milk powder, since color can be influenced by different factors such as processing conditions, storage, and the presence of Maillard reaction products. Regular monitoring of color helps ensure product consistency and detect potential quality issues (MILOVANOVIĆ et al., 2020).

Browning Index (BI)

The Browning Index (BI), which represents the intensity of the brown color in the samples, was determined based on the L , a , and b^* values from the color measurements. This index is commonly used to evaluate non-enzymatic browning, such as the Maillard reaction and the caramelization that can occur during processing and storage of dairy products (NASSER et al., 2017).

$$BI = \frac{[100(x - 0,31)]}{0,17} \quad (\text{Eq.10})$$

$$\text{With } x = \frac{(a^* + 1.750 \times L^*)}{(5.645 \times L^* + a^* - 3.012 \times b^*)} \quad (\text{Eq.11})$$

4.6 Functional Properties Analysis: Foam Capacity and Stability

The procedure described by ZHAO et al. (2022) was used to determine the foam capacity and stability of the milk powders. This method evaluates the ability of milk powders to form and maintain foam, which is important for applications in products. First, a 3 g sample of milk powder was dissolved in 100 mL of water to prepare the solution, which after was homogenized using an ultraturrax (Biovera, IKA T25 Digital, Rio de Janeiro, Brazil) at 10,000 rpm for 1 minute to generate foam. The volume of the resulting foam was immediately measured (V_0), and the foam volume was noted once more after a 30-minute period (V_{10}). The equations below were used to calculate foam capacity (FC) and foam stability (FS)

$$\text{Foam capacity} = \frac{V_0}{V} \times 100 \quad (\text{Eq.112})$$

$$\text{Foam stability} = \frac{V_{10}}{V_0} \times 100 \quad (\text{Eq.13})$$

4.7 Statistical Analysis

The full factorial design (2^3) was chosen for this research, which is designed with 3 variables and 2 levels for each one, 8 treatments were planned. As can be seen in Table 4, the variables and levels are the types of milk (whole and skimmed), evaporation (using open pan evaporator and rotary evaporator) and drying (spray and freeze drying). Table 4. shows statistical design with the variables and levels that were used in this research.

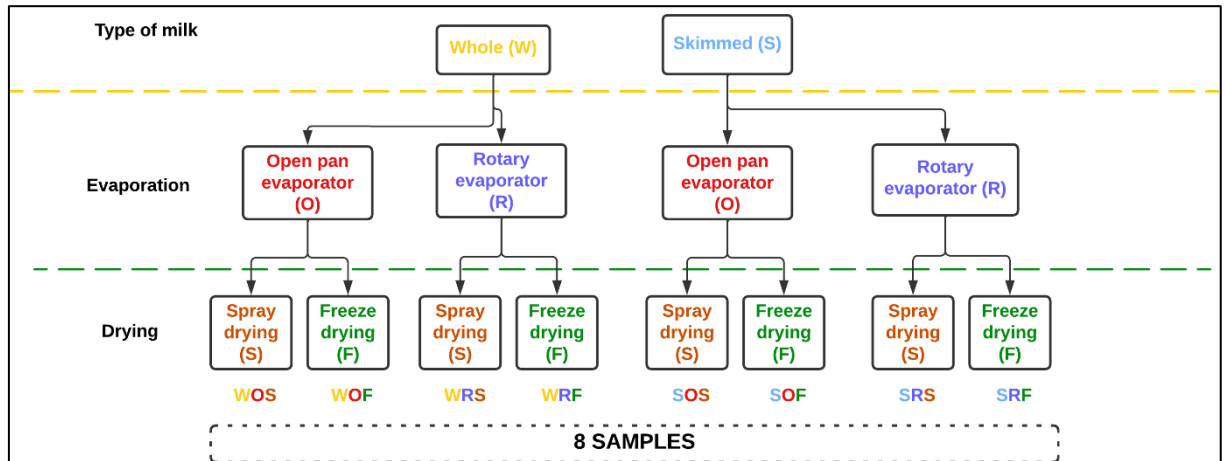
Table 4 -Full factorial design (2^3): variables and levels

| Variable | Levels | |
|--------------|---------------------|-------------------|
| Type of milk | Whole | Skimmed |
| Evaporators | Open pan evaporator | Rotary evaporator |
| Drying | Spray drying | Freeze drying |

Chemical composition, as well as physical, functional, and morphological properties, were considered dependent variables in the assessment. Each analysis was performed in triplicate, resulting in a total of 24 experimental runs, as seen in figure 6. Statistical analysis was carried out using Statistica 7 software. ANOVA was performed, followed by Tukey's test to conduct multiple comparisons. Both tests used the sum of squares (SS) residual as the error term.

Statistical significance was defined as a p-value less than 0.05. Mean \pm Standard Deviation ($n = 3$) is used to express the results, with different superscripts within the same row indicating statistically significant differences ($P < 0.05$).

Figure 6 - Full factorial design (2^3): variables and levels.



Source: the author

5. RESULTS AND DISCUSSION

5.1 Comparative Analysis of Chemical Composition in Liquid Milk and Milk Powder Samples

Table 5 presents the chemical composition of whole and skimmed milk, while Table 6 outlines the minimum composition requirements for both types of milk according to RTIQ

Table 5-Chemical composition of both whole and skimmed milk

| Type of milk | Fat (g/100 g) | Protein (g/100 g) | Moisture (g/100 g) | Ash (g/100 g) | Carbohydrates (g/100 g) |
|----------------|------------------------|------------------------|-------------------------|------------------------|-------------------------|
| Whole | 2,9±0,14 ^a | 6,06±0,12 ^a | 85,23±0,24 ^a | 0,84±0,05 ^a | 3,745±0,38 ^a |
| Skimmed | 0,58±0,04 ^b | 5,27±0,09 ^b | 87,04±0,31 ^b | 0,99±0,04 ^b | 6,27±0,15 ^b |

Mean ± Standard Deviation ($\times\pm\sigma$); N = 3 is used to present the results. Different letters within the same row show significant differences in the composition of the samples

Table 6- Minimum values for the chemical composition of whole and skimmed milk according to RTIQ.

| Type of milk | Fat (g/100 g) | Protein (g/100 g) | Moisture (g/100 g) | Carbohydrates (g/100 g) |
|----------------|---------------|-------------------|--------------------|-------------------------|
| Whole | 3,00 | 2,90 | 87,00 | 4,30 |
| Skimmed | 0,50 | 2,90 | 91,60 | 4,30 |

(REGULAMENTOS TÉCNICOS DE IDENTIDADE E QUALIDADE (RTIQ), 2018)

Table 5 presents the chemical composition of both whole and skimmed liquid milk and compares these values with those listed in Table 6. The fat content of both types of milk does not meet the minimum values recommended by the RTIQ, although the values are very close to the required minimum. For whole milk, the carbohydrate content (3.745 ± 0.38) is lower than the minimum value of 4.30 g/100g required by the RTIQ.

The chemical composition of the milk powder samples was compared to the values for whole and skimmed liquid milk. Table 7 shows the abbreviations used to identify each of the eight treatments analyzed.

Table 7. Abbreviations used to identify each of the eight treatments for the milk powder samples.

| Type of milk | Evaporator | Spray Technique |
|-----------------|------------------------|-----------------|
| W: Whole milk | O: Open pan evaporator | S: Spray dryer |
| S: Skimmed milk | R: Rotary evaporator | F: Freeze dryer |

Table 8-Chemical composition of milk powder samples

| Milk Powder | Fat (g/100 g) | Protein (g/100 g) | Moisture (g/100 g) | Ash (g/100 g) | Carbohydrates (g/100 g) |
|-------------|-------------------------|-------------------------|------------------------|-------------------------|-------------------------|
| WOS | 13,50±0,71 ^b | 24,79±2,28 ^b | 4,64±0,15 ^b | 13,18±0,2 ^c | 45,92±0,73 ^b |
| WOF | 19,50±0,71 ^a | 21,89±3,44 ^c | 4,36±0,31 ^b | 16,87±1,09 ^b | 36,72±0,69 ^c |
| WRS | 19,50±0,71 ^a | 22,76±0,16 ^c | 3,54±0,12 ^c | 15,79±0,92 ^b | 35,55±3,35 ^e |
| WRF | 18,25±0,35 ^a | 23,35±0,29 ^b | 3,65±0,24 ^c | 17,11±0,35 ^a | 39,09±2,99 ^c |
| SOS | 4,75±0,35 ^d | 29,86±0,62 ^a | 5,34±0,74 ^a | 18,81±1,45 ^a | 43,81±2,42 ^b |
| SOF | 5,75±0,35 ^c | 29,93±0,22 ^a | 3,05±0,08 ^c | 10,95±0,77 ^d | 51,65±1,34 ^a |
| SRS | 6,75±0,35 ^c | 29,25±2,36 ^a | 3,47±0,17 ^c | 9,66±1,62 ^d | 50,68±1,88 ^a |
| SRF | 6,10±0,14 ^c | 28,19±0,38 ^a | 3,22±0,14 ^c | 16,34±1,65 ^b | 44,41±1,91 ^d |

W: whole milk; S: skimmed milk; O: open pan evaporator; R: rotary evaporator; S: spray dryer; F: freeze dryer
Mean ± Standard Deviation ($\times\pm\sigma$); N = 3 is used to present the results. Different letters within the same row show significant differences in the composition of the samples

Table 8 illustrate changes in the chemical composition of liquid milk and milk powder following the evaporation and drying. Whole milk powders (WOF, WRS, WRF) exhibit the highest fat content, ranging from 18.25% to 19.5%, while skimmed milk powders contain significantly less fat, between 4.75% and 6.75%. On the other hand, skimmed milk powders have the highest protein content, between 28.19% and 29.93%, due to the removal of fat, which increases the relative concentration of protein. Whole milk powders, in contrast, have lower protein content than skimmed milk powders.

All milk powders have a significantly lower moisture content compared to liquid milk, which is essential to enhancing the shelf life of powdered milk products, as noted by LANGOVÁ; ŠTENCL (2014). However, the SOS sample has a moisture content of $5.34 \pm 0.74\%$, which exceeds the maximum limit set by the REGULAMENTOS TÉCNICOS DE IDENTIDADE E QUALIDADE (RTIQ) (2018), which requires milk powder to have less than 5.00% moisture (TERESA et al., 2018). This discrepancy could be due to the spray dryer

employs hot air to rapidly evaporate water from the milk, which not remove all of the moisture (DESHWAL et al., 2020).

Skimmed milk powders also have a higher ash content (up to 18.81%) than whole milk powders, which range from 13.18% to 17.11%, as the removal of fat tends to concentrate minerals. Finally, whole milk powders (WOF, WRS) have a lower carbohydrate content compared to skimmed milk powders (SOF, SRS).

Studies have shown that variations in the chemical composition of milk powder can influence its functional properties. For instance, ZOUARI et al. (2020) stated that higher fat content can lead to fat accumulation on the surface of the particles, making them hydrophobic. This reduces key properties such as wettability, flowability, and dispersibility, particularly in samples with a high-fat content like WOS, WRS, WOF, and WRF.

Several researchers have explained that during the production of milk powder, proteins may denature, exposing hydrophobic regions, which leads to aggregation and reduced solubility in water (SUDHARSHAN et al., 2014). ANEMA (2019) mentioned that the major whey proteins, β -lactoglobulin and α -lactalbumin, experience notable denaturation. It happens only when milk is heated beyond 70°C and KELLY; O'CONNELL; FOX (2016) explained that when milk is preheated above 75°C at a pH of 6.5, β -lactoglobulin begins to denature. As the temperature rises above 90°C, this denaturation becomes more extensive, and α -lactalbumin can also start to denature.

The increase in temperature can influence how whey proteins interact, favoring whey protein–whey protein interactions and leading to more extensive denaturation. Additionally, if evaporated concentrate is held at temperatures above 60°C for extended periods which can cause the aggregation of casein micelles and raise the viscosity of the concentrate (KELLY; O'CONNELL; FOX, 2016). This phenomenon could affect all samples, but it is more pronounced in those with higher concentrations, such as SOS, SRS, SOF, and SRF.

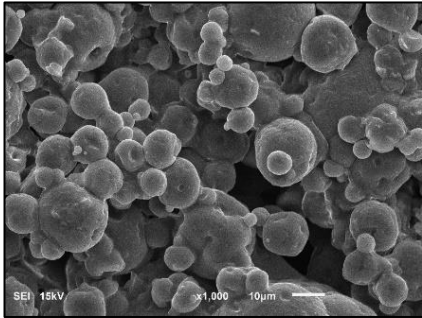
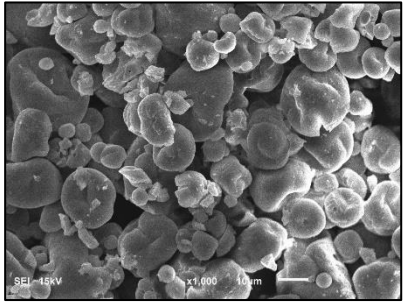
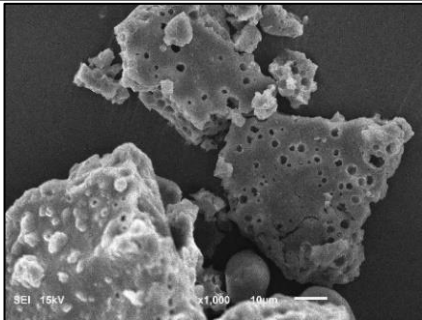
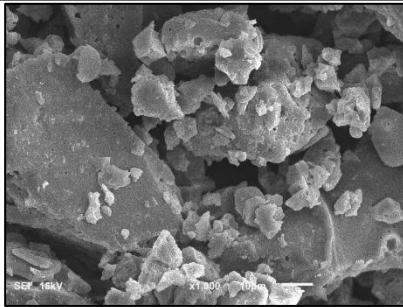
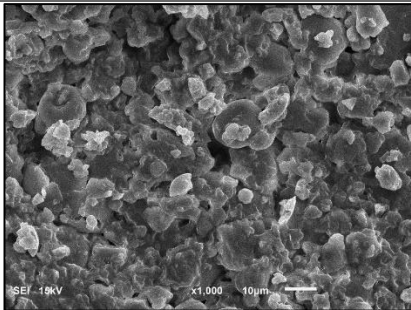
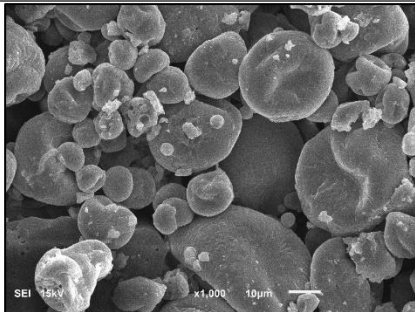
Moisture plays an important role in milk powder, as it directly affects the final product's shelf life and influences amorphous powders' transition and crystallization behavior. These factors impact the powder's flowability, stickiness, caking, and storage stability (FOURNAISE et al., 2020; HUPPERTZ; GAZI, 2016).

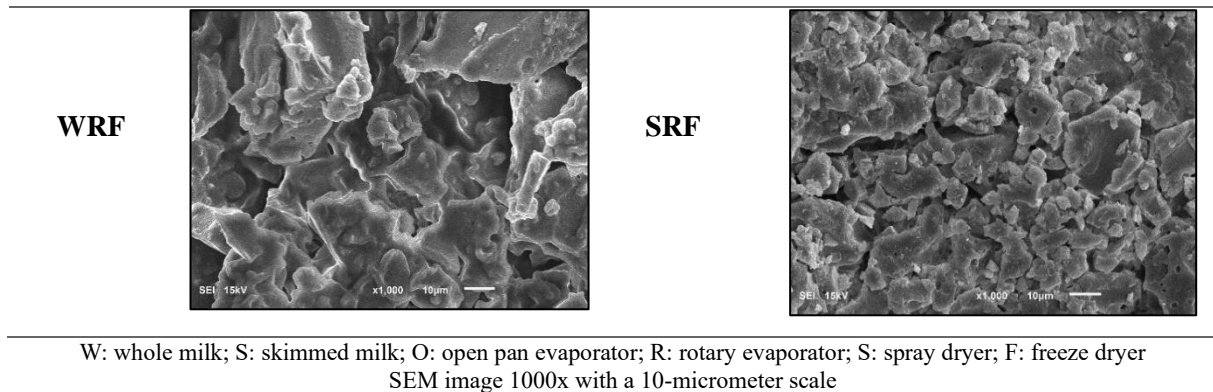
Overall, the chemical composition of milk and milk powder is key to their physical and functional properties. Changes in the percentages of fat, protein, and carbohydrates can greatly affect milk powder properties, which will be discussed further in Section 5.3.

5.2 Microstructure of Milk Powder

Table 9 presents the microstructure of milk powder. The impact of the concentration method and milk type on the microstructure was not clearly observed. However, the drying method significantly influenced morphology. Spray-dried samples have smooth surfaces and spherical particles, while freeze-dried samples appear rough and porous.

Table 9-Microstructure and diameter of milk powder.

| Milk Powder | SEM image | Milk Powder | SEM image |
|-------------|---|-------------|---|
| WOS |  | SOS |  |
| WOF |  | SOF |  |
| WRS |  | SRS |  |



This difference can be explained by the drying technique. In spray drying, the nozzle evenly disperses liquid droplets into the drying chamber, leading to the creation of spherical particles. In contrast, freeze drying preserves the original structure of the particles, resulting in minimal volume change and the formation of rough, porous particles (DESHWAL et al., 2020; ZAFISAH et al., 2018).

These results are in accordance with DESHWAL et al. (2020), who observed that freeze-dried powders have flake-like structure, whereas spray-dried powders form clusters of individual spherical particles. Besides, ZAFISAH et al. (2018) observed that freeze-dried powders have irregular shapes and skeleton-like structures with increased porosity due to the freeze-drying.

Overall, the drying conditions can affect the microstructure of milk powder, which, in turn, may influence properties such as solubility, rehydration, and flowability. These findings will be discussed in the following section.

5.3 Flowability, Compressibility, and Packing Properties: Bulk Density, Compact Density, Carr's Index (CI) and Hausner Ratio (HR)

Table 10 presents the bulk density, compact density, Carr's Index (CI), and Hausner Ratio (HR) values of milk powder samples. Flowability refers to how easily a powder flows, while cohesiveness describes the tendency of particles to stick together. Higher cohesiveness results in poorer flowability (PUGLIESE et al., 2017). In the same context, lower CI and HR values indicate lower cohesiveness and better flowability (SEO, 2022).

Table 10-Bulk density, compact density, Carr's Index (CI), and Hausner Ratio (HR) values of milk powder samples

| Milk powder | Bulk Density (g/mL) | Compact density (g/mL) | Carr's Index (CI) (%) | Hausner Ratio (HR) |
|-------------|------------------------|------------------------|-------------------------|------------------------|
| WOS | 0,44±0,00 ^a | 0,50±0,01 ^b | 12,61±0,39 ^c | 1,14±0,01 ^a |
| WOF | 0,27±0,05 ^b | 0,40±0,00 ^a | 25,65±0,31 ^b | 1,55±0,35 ^b |
| WRS | 0,33±0,02 ^b | 0,42±0,02 ^a | 17,34±2,11 ^a | 1,28±0,12 ^a |
| WRF | 0,33±0,00 ^b | 0,39±0,00 ^a | 14,80±0,76 ^a | 1,17±0,01 ^a |
| SOS | 0,43±0,04 ^a | 0,56±0,01 ^c | 25,40±1,05 ^b | 1,30±0,08 ^a |
| SOE | 0,39±0,02 ^a | 0,48±0,01 ^d | 20,42±0,02 ^d | 1,30±0,07 ^a |
| SRS | 0,40±0,02 ^a | 0,50±0,02 ^b | 24,80±2,56 ^b | 1,38±0,1 ^a |
| SRF | 0,40±0,02 ^a | 0,51±0,02 ^b | 19,20±1,6 ^d | 1,27±0,06 ^a |

W: whole milk; S: skimmed milk; O: open pan evaporator; R: rotary evaporator; S: spray dryer; F: freeze dryer
Mean ± Standard Deviation ($\times\pm\sigma$); N = 3 is used to present the results. Different letters within the same row show significant differences in the composition of the samples

5.3.1 Bulk and Compact Density

Table 10 indicates that both the compact and bulk densities of skimmed milk powder are significantly higher ($p < 0.05$) than whole milk powder densities. This result is due to the lack of fat and the higher protein content in skimmed milk powder. PUGLIESE et al. (2017) explained that fat has a lower density than protein; therefore, as fat content increases, the proportion of protein per unit of mass decreases.

The statistical analysis showed that both concentration and drying caused significant differences ($p < 0.05$) between treatments. Samples from the open pan evaporator and spray dryer (WOS: 0.44 ± 0.00 g/mL; 0.50 ± 0.01 g/mL and SOS: 0.43 ± 0.04 g/mL; 0.56 ± 0.01 g/mL) had higher bulk and compact densities, respectively.

This finding can be attributed to two reasons. Firstly, evaporation at 100.33°C led to the denaturation of proteins such as β -lactoglobulin and α -lactalbumin. Additionally, it promoted whey protein–whey protein interactions, which facilitated the clustering of casein micelles. This aggregation resulted in the formation of denser particle clusters, increasing the overall density of the milk powder (KELLY; O'CONNELL; FOX, 2016).

Secondly, the spherical particles formed during spray drying tend to have rough surfaces, which can contribute to an increase in density. This is due to their solid structure and small, spherical shape (WOS: 14.02 ± 0.97 μm and SOS: 13.6 ± 0.71 μm), as shown in Table

11. These characteristics allow for a higher mass to be packed into the same volume (DESHWAL et al., 2020).

Table 11- Diameter of Milk Powder Particles

| Milk powder | Diameter (μm) |
|--------------------|--|
| WOS | 14,02 \pm 0,97 ^a |
| WOF | 124,57 \pm 13,06 ^c |
| WRS | 13,07 \pm 1,4 ^a |
| WRF | 56,42 \pm 1,91 ^d |
| SOS | 13,6 \pm 0,71 ^a |
| SOF | 37,1 \pm 1,51 ^c |
| SRS | 19,79 \pm 1,15 ^b |
| SRF | 17,85 \pm 1,8 ^b |

W: whole milk; S: skimmed milk; O: open pan evaporator; R: rotary evaporator; S: spray dryer; F: freeze dryer
Mean \pm Standard Deviation ($\times\pm\sigma$); N = 3 is used to present the results. Different letters within the same row show significant differences in the composition of the samples

In contrast, samples with lower bulk and compact densities (WOF: 0.27 \pm 0.05 g/mL, 0.40 \pm 0.00 g/mL; and SOF: 0.39 \pm 0.02 g/mL, 0.48 \pm 0.01 g/mL) exhibited particles with a porous, sheet-like structure, as shown in Table 9. This morphology is associated with voids trapped within the porous structure, which maintain a similar volume but contain mass, resulting in lower density. This aligns with findings from DESHWAL et al. (2020) and ZAFISAH et al. (2018), who observed the same appearance in their freeze-dried milk powders, while spray-dried milk powder showed regular-shaped particles.

Porosity is another parameter that affects the density of milk powder. As an example, the sample with the highest density (SOS) exhibits lower porosity, as seen in Table 9. High compact density is associated with lower porosity and is closely linked to storage efficiency, as powders with higher bulk densities occupy less space (BALDELLI et al., 2022).

5.3.2 Carr's Index (CI) and Hausner Ratio (HR)

Table 10 presents the effects of the type of milk, evaporation, and drying methods on the Carr's Index (CI) and Hausner Ratio (HR), as indicated by statistical analysis. In general, CI is higher in skimmed powder, while Hausner Index (HR) values were similar for all milk powder types. An exception is WOF powder, which exhibited the highest Carr Index (CI) value and significantly higher Hausner Ratio (HR), suggesting poor flowability and high cohesiveness. This

result can be attributed to its high fat content (19.5 ± 0.71 g/100 g), which increases the adhesion and cohesion forces between particles (SILVA; O'MAHONY, 2017).

On the other hand, the evaporation and drying methods did not significantly influence CI and HR, particularly in terms of powder flowability. However, slight differences were noted between the CI values of spray-dried and freeze-dried samples.

Whole milk powder produced by open-pan evaporation and spray drying (WOS sample) exhibited a lower Carr Index (12.61 %) and Hausner Ratio (1.14), indicating fair flowability. Both CI and HR are used to classify the flow properties of milk powder. According to PUGLIESE et al. (2017) HR values below 1.34 and CI values under 15 % correspond to excellent to good flowability, which is essential for efficient packaging.

This result may be attributed to the lower porosity of the WOS sample, which reduces the number of voids available for particle agglomeration, as observed in Table 9. Although its reduced particle dimensions (14.02 ± 0.97 μm) are generally associated with reduced flowability, high-fat milk powders like WOS are less affected. This is likely due to the fat content, which decreases particle adhesion and enhances flow properties, according to SUHAG; KELLIL; RAZEM (2024).

5.4 Stability and Reconstitution Properties: Water Activity, Hygroscopicity, Solubility and Wettability

Table 12 presents the values for water activity, hygroscopicity, solubility, and wettability, which are associated with stability and reconstitution properties of the milk powder samples.

Table 12-Values for water activity, hygroscopicity, solubility, and wettability of milk powder samples.

| Milk Powder | Water activity (a_w) | Hygroscopicity (g/100 g) | Solubility (%) | Wettability (min/g) |
|-------------|--------------------------|--------------------------|--------------------|---------------------|
| WOS | $0,36 \pm 0,01^c$ | $8,29 \pm 0,15^b$ | $64,84 \pm 5,84^b$ | $58,69 \pm 0,00^c$ |
| WOF | $0,20 \pm 0,01^c$ | $9,56 \pm 0,16^a$ | $69,67 \pm 2,94^b$ | $231,37 \pm 8,99^d$ |
| WRS | $0,33 \pm 0,01^d$ | $8,16 \pm 0,22^b$ | $60,59 \pm 2,95^b$ | $164,02 \pm 2,79^e$ |
| WRF | $0,12 \pm 0,01^a$ | $8,19 \pm 0,09^b$ | $65,2 \pm 10^b$ | $46,16 \pm 0,95^f$ |
| SOS | $0,42 \pm 0,00^b$ | $11,71 \pm 0,29^c$ | $82,85 \pm 1,21^a$ | $2,77 \pm 0,19^g$ |
| SOF | $0,14 \pm 0,01^a$ | $10,49 \pm 0,27^a$ | $80,67 \pm 7,32^a$ | $8,01 \pm 0,07^a$ |
| SRS | $0,13 \pm 0,01^a$ | $9,87 \pm 0,16^a$ | $94,71 \pm 0,58^c$ | $7,76 \pm 0,64^a$ |

| | | | | |
|------------|------------------------|-------------------------|-------------------------|-------------------------|
| SRF | 0,15±0,00 ^a | 10,29±0,96 ^a | 78,95±3,95 ^d | 10,74±0,40 ^b |
|------------|------------------------|-------------------------|-------------------------|-------------------------|

W: whole milk; S: skimmed milk; O: open pan evaporator; R: rotary evaporator; S: spray dryer; F: freeze dryer
Mean ± Standard Deviation ($\times\sigma$); N = 3 is used to present the results. Different letters within the same row show significant differences in the composition of the samples

5.4.1 Water activity and Hygroscopicity

Table 12 presents the water activity (WA) values and hygroscopicity of the milk powder samples. The type of milk, concentration, and drying significantly influenced ($p < 0.05$) the water activity. The highest water activity values were observed in whole and skimmed milk powders produced by open pan evaporator and spray dryer (WOS: 0.36 ± 0.01 ; SOS: 0.42 ± 0.00). These samples also had the highest moisture content (WOS: 4.64 ± 0.15 g/100 g; SOS: 5.34 ± 0.74 g/100 g), as shown in Table 8.

The open pan evaporator may lead to uneven moisture removal, causing certain areas of the milk to retain higher residual water content. In contrast, the rotary evaporator allows for a more efficient and uniform removal of water, resulting in a higher concentration of solids before drying. As a result, the more concentrated samples contain less free water, leading to lower water activity in the final product (WHITEPAPER, 2021).

Spray-dried powders typically have a higher moisture content compared to freeze-dried samples, which results in greater water activity. This is because the spray drying uses hot air to rapidly evaporate water (DESHWAL et al., 2020), which can leave a higher moisture content in the sample (LEE et al., 2023). Moreover, spray drying can produce smaller powder particles (13.07 ± 1.4 μm to 19.79 ± 1.15 μm), as seen in table 11. These smaller particles possess a greater surface area, which increases their exposure to humidity (PUGLIESE et al., 2017).

The drying technique significantly influenced hygroscopicity. The samples produced with freeze dryer presented higher hygroscopicity likely porous and amorphous structure powder created by the sublimation. Amorphous lactose is highly hygroscopic leading to higher hygroscopicity compared to spray-dried samples (HAQUE; ROOS, 2005). It was also observed that skimmed milk powder exhibited higher hygroscopicity values. This result can be attributed to higher lactose content (43.81 ± 2.42 g/100 g to 51.65 ± 1.34 g/100 g) and a relatively high protein percentage (28.19 ± 0.38 g/100 g to 29.93 ± 0.22 g/100 g) as seen in table 8.

The presence of amorphous lactose tends to absorb moisture from the environment. The uptake of moisture can trigger lactose crystallization, releasing free water and subsequently increasing water activity and hygroscopicity (HUPPERTZ; GAZI, 2016). Moreover, proteins

may also contribute moderately to moisture absorption (LEE et al., 2023) as seen in the SOS sample. This sample exhibited the highest hygroscopicity (11.71 ± 0.29 g/100 g) and relatively high protein (29.86 ± 0.6 g/100 g) and lactose percentages (43.81 ± 2.42 g/100 g).

Samples with the highest hygroscopicity can absorb the most moisture from the environment, which may affect the solubility of the milk powder. If the powder absorbs excess moisture, it can form clumps (MAIDANNYK et al., 2020). Additionally, HO et al. (2019) highlighted that water activity is a key factor in controlling the stability of milk powder during storage.

5.4.2 Solubility and Wettability Time

Table 12 presents the solubility and wettability times of the milk powders. According to statistical analysis, the type of milk exerts the greatest influence ($p < 0.05$) on these properties. Skimmed milk powder shows higher solubility percentages (78.95 ± 3.95 % to 94.71 ± 0.58 %) and shorter wettability times (2.77 ± 0.19 min/g to 10.74 ± 0.4 min/g). This can be attributed to its higher protein content and lower fat content.

Skimmed milk powder has a lower fat content (4.75 ± 0.35 g/100 g to 6.75 ± 0.35 g/100 g), resulting in a higher proportion of proteins and lactose, which dissolve more easily in water. In contrast, milk fat can act as a barrier to solubility (MOHAN et al., 2020). Additionally, casein and whey proteins dissolve more readily in water, further enhancing solubility (RUPP; MOLITOR; LUCEY, 2018). Similar studies, such as those by DESHWAL et al. (2020) reported higher wettability times in whole milk powder, attributing this to the lipophilic nature of milk fat.

Another critical factor affecting solubility is particle size. For example, the SOS sample exhibited high solubility (82.85 ± 1.21 %) and the lowest wettability time (2.77 ± 0.19 min/g), which can be linked to its smaller particle diameter (13.6 ± 0.71 μm), as seen in Tables 12 and 11. Smaller particles increase the surface area of contact between solids and liquids, potentially enhancing solubility in water (BALDELLI et al., 2022).

Neither concentration nor drying method had a statistically significant effect on solubility or wettability times, as shown in the Pareto Diagram of Standardized Effects Related to solubility in the figures 7 and 8. JI et al. (2016) noted that both solubility and wettability are essential for rehydration properties; however, lower solubility or wettability can lead to clumping, poor texture, and ultimately affect both product quality and consumer satisfaction.

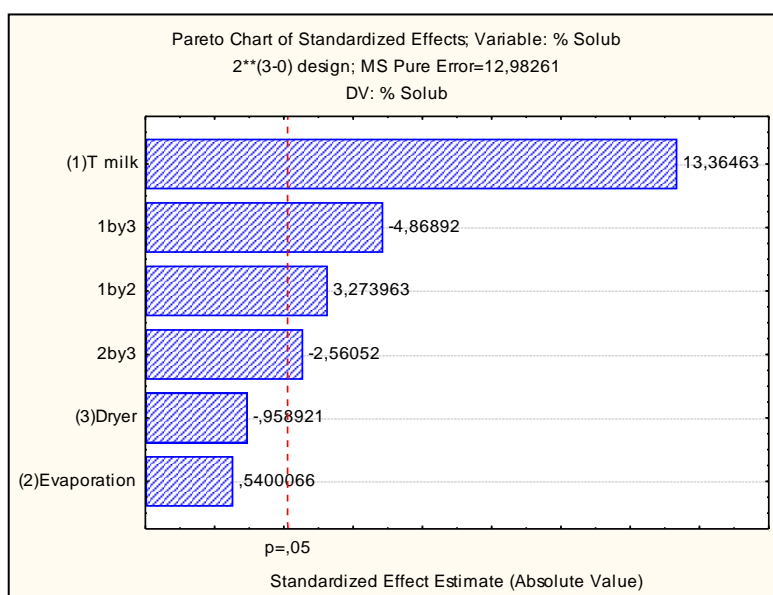


Figure 7. Pareto Diagram of Standardized Effects Related to solubility.

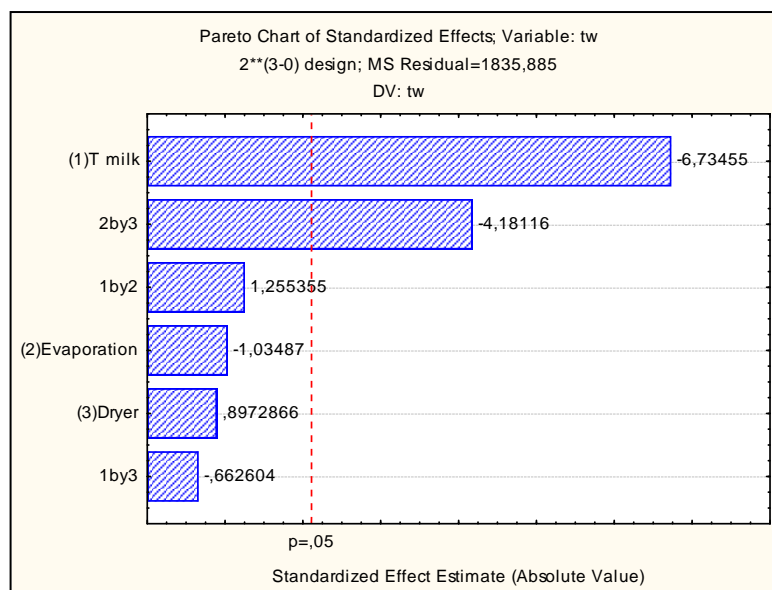


Figure 8. Pareto Diagram of Standardized Effects Related to wettability time

5.5 Color Properties: L^* , a^* , b^* and Browning Index (BI)

Table 13 shows the color parameters (L , a , b^* , and Browning Index or BI) for the milk powder samples, along with values obtained from the literature (PONNAL et al., 2021).

Table 13-Color parameters: L*, a*, b* and Browning Index (BI) of milk powder

| Milk Powder | L* | a* | b* | BI |
|-------------|-------------------------|-------------------------|-------------------------|-------------------------|
| WOS | 91,46±0,2 ^a | -0,16±0,09 ^a | 23,12±0,76 ^b | 28,3±1,05 ^b |
| WOF | 91,68±0,17 ^a | -0,14±0,08 ^a | 21,54±0,16 ^c | 26,02±0,27 ^b |
| WRS | 92,99±0,34 ^b | -0,92±0,11 ^b | 13,20±0,17 ^d | 14,2±0,24 ^a |
| WRF | 89,98±0,15 ^c | -1,15±0,11 ^b | 22,99±0,45 ^b | 28,65±0,72 ^b |
| SOS | 91,79±0,12 ^a | -0,36±0,15 ^a | 20,40±0,38 ^a | 28,16±3,37 ^b |
| SOF | 91,64±0,33 ^a | -0,18±0,22 ^a | 21,05±0,07 ^a | 29,26±3,39 ^b |
| SRS | 92,86±0,28 ^b | -2,33±0,13 ^d | 20,44±0,55 ^a | 26,27±2,93 ^b |
| SRF | 92,23±0,16 ^b | -1,61±0,07 ^c | 20,40±0,06 ^a | 26,98±3,34 ^b |
| *WMP-B | 96.01 ± 0.54 | -1.74 ± 0.31 | 14.45 ± 0.55 | 14,56±0,11 |
| *SMP-B | 96.94 ± 0.56 | -2.32 ± 0.14 | 11.12 ± 1.32 | 10,05±0,20 |

W: whole milk; S: skimmed milk; O: open pan evaporator; R: rotary evaporator; S: spray dryer; F: freeze dryer
Mean ± Standard Deviation ($\times\pm\sigma$); N = 3 is used to present the results. Different letters within the same row show significant differences in the composition of the samples *WMP-B: Color values of whole milk powder reported in the literature.

*SMP-B: Color values of skimmed milk powder reported in the literature

5.5.1 Color Parameters: L*, a* and b*

L* (Lightness)

The type of milk, evaporator method, and drying technique had a statistically significant effect on the L* color parameter, which is measured on a scale from 0 (black) to 100 (white) by the lightness. The eight samples produced in this study exhibited lower L* values ($89,98 \pm 0,15$ to $92,86 \pm 0,28$) than those reported in the literature (WMP-B: 96.01 ± 0.54 and SMP-B: 96.94 ± 0.56), indicating that they are darker or less white. This difference may be attributed to the higher temperatures used during concentration and drying, which can promote Maillard reactions (LEE et al., 2023). Additionally, variations in particle size and composition could further influence the lightness of milk powder (DESHWAL et al., 2020).

The WRS and SRS samples exhibited the highest L* values (WRS: 92.99 ± 0.34 and SRS: 92.86 ± 0.28) as seen in table 13, suggesting that rotary evaporation, which operates at a relatively low temperature (60°C), provides a gentle thermal treatment that helps preserve the natural color and quality of milk powder (MAURÍCIO et al., 2021). Moreover, the increased lightness (L*) observed in these treatments is strongly associated with their smaller particle diameters (WRS: $13.07 \pm 1.4 \mu\text{m}$ and SRS: $19.79 \pm 1.15 \mu\text{m}$, respectively) as seen in table 11.

Fewer particles disperse light more uniformly, resulting in a clearer appearance (PONNAL et al., 2021).

In contrast, the WRF sample exhibited the lowest L^* value (89.98 ± 0.15), indicating a darker color. This can be explained by its larger particle size ($56.42 \pm 1.91 \mu\text{m}$) (MILOVANOVIC et al., 2020). According to DESHWAL et al. (2020), the whiteness of milk is primarily influenced by smaller particle sizes and lower carotene content. Additionally, the freeze-drying may have affected the color of the milk powder due to oxidation of lipids and proteins, which can lead to color changes during extended freeze-drying times (COŞKUN et al., 2024).

***a** (Green-Red Spectrum)**

According to the statistical analysis, the type of milk, evaporator method, and drying technique had a statistically significant effect on a^* color parameter. Positive values show a redder tone, while negative values show a greener tone.

The samples produced in this study exhibited higher a^* value (less green) (-0.16 ± 0.09 to -2.33 ± 0.13) compared to those reported in the literature (WMP-B: -1.74 ± 0.31 and SMP-B: -2.32 ± 0.14). The difference is probably caused by the higher temperatures used during concentration and drying, which may have triggered Maillard reactions (LEE et al., 2023) or protein denaturation (ZHOU; LANGRISH, 2022), leading to a shift towards less green (higher a^* values).

The SRS sample exhibited the most negative a^* value (-2.33 ± 0.13), as shown in Table 13, making it the greenest sample. This can be explained by the fact that skim milk powder does not reflect the full spectrum of light wavelengths as effectively as whole milk powder (DESHWAL et al., 2020; PUGLIESE et al., 2017). The lack of fat in skim milk leads to increased light absorption by green compounds, such as riboflavin (PONNAL et al., 2021). Additionally, a more negative a^* value is associated with the rotary evaporation, as it provides a mild thermal treatment that helps preserve the natural color stability (MAURÍCIO et al., 2021).

***b** (Yellow-Blue Spectrum)**

The evaporator method and the drying technique had a statistically significant effect on the b color parameter, as indicated by the statistical analysis. However, the type of milk did not have a significant impact. The b^* parameter, which represents the yellowness (positive values)

and blueness (negative values) of milk powder, is shown in Table 13. Higher b^* values indicate a more intense yellow tone in the sample.

The samples produced in this study exhibited higher b^* values (13.20 ± 0.17 to 23.12 ± 0.76), indicating greater yellowness compared to the values reported in the literature (WMP-B: 14.45 ± 0.55 and SMP-B: 11.12 ± 1.32). This difference could be attributed to the heat treatments applied during processing, which may have influenced pigment stability and Maillard reaction products (DESHWAL et al., 2020).

The WOS and WRF samples showed higher b^* values (23.12 ± 0.76 and 22.99 ± 0.45), meaning they are more yellow, as seen in Table 13. This finding can be explained by the evaporator equipment used in each treatment: the WOS sample was produced using an open pan evaporator at $100\text{ }^\circ\text{C}$, while the WRS sample was produced with a rotary evaporator at $60\text{ }^\circ\text{C}$. Thus, the higher temperature in both treatments could have produced a yellower powder, (SULIEMAN et al., 2015). Moreover, whole milk, which has a higher fat content, can retain more carotenoids, resulting in a more pronounced yellow color (MILOVANOVIĆ et al., 2020; DESHWAL et al., 2020).

Overall, the variations in milk powder color can be attributed to the interaction of multiple factors, including heat treatments that trigger Maillard reactions, changes in particle size, and the concentration of pigments such as riboflavin and carotenoids (MILOVANOVIĆ et al., 2020; DESHWAL et al., 2020). The color of milk powder not only influences its visual appeal to consumers and industries but can also serve as a quality indicator (SULIEMAN et al., 2015).

5.5.2 Browning Index (BI)

As shown in figure 9, the type of milk, evaporator method, and drying technique did not have a statistically significant effect on BI. However, the eight samples produced in this study exhibited higher BI values (14.2 ± 0.24 to 29.26 ± 3.39) compared to those reported in the literature (WMP-B: 14.56 ± 0.11 and SMP-B: 10.05 ± 0.20). This difference could be attributed to greater heat exposure (DESHWAL et al., 2020) such as spray drying at elevated temperatures ($150\text{ }^\circ\text{C}$) or open-pan evaporation ($100,33\text{ }^\circ\text{C}$), which may accelerate Maillard reactions (RYAN; O'REGAN; FITZGERALD, 2023). Additionally, prolonged thermal processing during drying could further contribute to browning in the samples (COŞKUN et al., 2024).

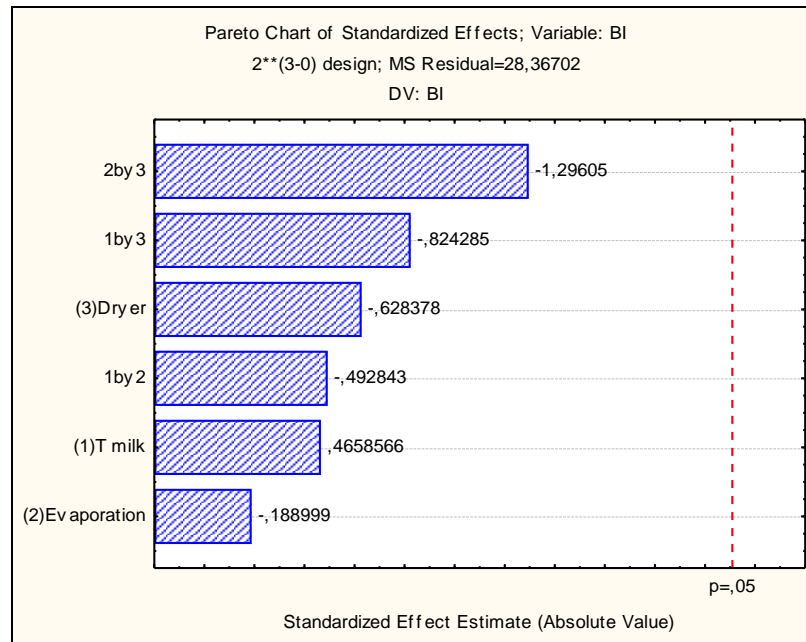


Figure 9. Pareto Diagram of Standardized Effects Related to Browning Index.

Although the type of milk, evaporator method, and drying technique did not have a statistically significant effect on BI, variations in BI values were observed, which could be related to color parameters (L^* , a^* , and b^*). The WRS sample exhibited the lowest BI values (14.2 ± 0.24), indicating a lighter brown tone. This sample also showed the lightest color ($L^* = 92.99 \pm 0.34$) and the lowest b^* value (13.20 ± 0.17), suggesting a less yellow tone, as seen in the table 13. Moreover, this sample was the closest to the values reported in the literature. The use of a rotary evaporator may explain this result, as it uses moderate heat and vacuum pressure to remove water, leading to milder thermal exposure and reduced browning (DESHWAL et al., 2020; MAURÍCIO et al., 2021).

The sample with the highest BI (29.26 ± 3.39) was the SOF, which exhibited both relative values: a high b^* value (21.05 ± 0.07) and a low L^* value (91.64 ± 0.33), indicating a darker sample with a tendency toward a browner tone. This result may be linked to the use of the open pan evaporator, which applies high heat for extended periods, potentially promoting intense Maillard reactions and causing more browning (KERR, 2019). Additionally, skimmed milk powders can trigger Maillard reactions during high-temperature treatments due to their high lactose content (ROMO et al., 2024). In fact, the SOF sample has the highest lactose content (51.65 ± 1.34) as seen in table 8.

Regarding the drying technique, high BI values were observed in samples processed using both spray drying and freeze drying. The browning observed in spray-dried samples is

probably because of the high temperatures applied during the process, which range from 130°C to 170°C (HO et al., 2019). On the other hand, although freeze drying operates at low temperatures and relies on sublimation, which can almost completely prevent browning (COŞKUN et al., 2024), MICHALSKA et al., (2019) demonstrated that the prolonged drying times and extremely low moisture conditions in freeze drying can lead to the formation of hydroxymethylfurfural (HMF), a compound associated with browning in milk powder.

Overall, higher BI values indicate greater browning in the milk powder, which can be attributed to several factors, including the type of milk used (e.g., skimmed milk), high temperatures during processes like evaporation and spray drying, and extended drying times at lower temperatures, as seen in freeze drying. The color of dairy powders is a crucial factor in determining consumer acceptance of the final product, and the browning index (BI) is a quantitative measure used to assess the level of browning in food products (NASSER et al., 2017).

5.6 Foaming Properties

5.6.1 Foam Capacity and Stability

The foaming properties are shown in Table 14. Based on statistical analysis, the type of milk had a statistically significant effect ($p < 0.05$) on foaming ability. However, other factors, such as evaporation and drying methods were not statistically significant.

Table 14-Foam capacity and stability of milk powder samples.

| Milk Powder | Foaming Capacity (%) | Foaming Stability (%) |
|--------------------|-----------------------------|------------------------------|
| WOS | 102,2±1,11 ^a | 99,65±0,61 ^b |
| WOF | 101,67±0,57 ^a | 99,64±0,62 ^b |
| WRS | 101,09±0,01 ^a | 100,18±0,31 ^c |
| WRF | 102,21±0,01 ^a | 99,29±0,61 ^b |
| SOS | 104,06±0,66 ^b | 98,22±0,78 ^a |
| SOF | 103,75±1,33 ^b | 101,65±0,55 ^d |
| SRS | 104,12±1,36 ^b | 100,36±0,63 ^c |
| SRF | 104,83±0,63 ^b | 100,18±0,31 ^b |

W: whole milk; S: skimmed milk; O: open pan evaporator; R: rotary evaporator; S: spray dryer; F: freeze dryer
Mean ± Standard Deviation ($\times\pm\sigma$); N = 3 is used to present the results. Different letters within the same row show significant differences in the composition of the samples

The SOS, SOF, SRS, and SRF samples exhibited the highest foam capacity, ranging from $103.75 \pm 1.33\%$ to $104.83 \pm 0.63\%$, which is linked to their high protein content (28.19 ± 0.38 g/100 g to 29.93 ± 0.22 g/100 g), as shown in Tables 14 and 8. Several studies have demonstrated that high protein content enhances the foaming stability of milk powder due to the amphiphilic nature of proteins, allows them to create a stable layer around air bubbles., reducing bubble coalescence and maintaining foam stability (HO et al., 2024). Additionally, proteins like casein and whey can create elastic, collapse-resistant networks around air bubbles, further enhancing foam stability (HO; BHANDARI; BANSAL, 2022).

Table 14 shows the highest foam stabilities in SOF, SRS, and SRF samples ranging from $101,65 \pm 0,55$ % to $98,22 \pm 0,78$ %, which may be related to the high protein content, according to SHILPASHREE et al. (2015). In contrast, the high content in whole milk power could decrease the foaming capacity due to fat molecules can disrupt the film created by protein around the air bubbles. Moreover, both fat and protein can compete each other. Fat tends to migrate faster to the interface which can stabilize the air bubbles (HO; BHANDARI; BANSAL, 2020).

On the other hand, the interactions between milk type, evaporation equipment, and drying technique have a significant effect on foam stability. For instance, the interaction between milk type and drying method positively affected foam stability, indicating that combining certain milk types with specific drying methods can enhance stability. In contrast, the interaction between evaporator type and dryer type had a significant negative effect, suggesting that certain combinations of these processes can reduce foam stability, as seen in figure 10.

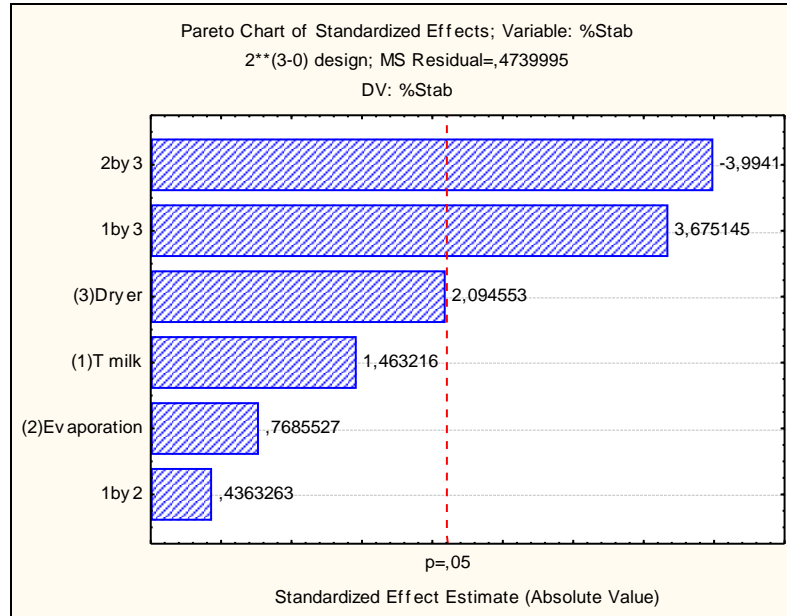


Figure 10. Pareto Diagram of Standardized Effects Related to Foaming Stability

Foaming is a key property of milk powder, primarily influenced by protein and fat content. The amphiphilic nature of proteins enables them to form elastic, collapse-resistant films around air bubbles, enhancing stability. In contrast, high fat content can disrupt these films, reducing foaming capacity. The balance between protein and fat determines overall foam formation and stability (HO et al., 2024; HO; BHANDARI; BANSAL, 2022).

6. CONCLUSION

This study investigated the effects of milk type, evaporation equipment, and drying techniques on the properties of milk powder, categorized into the following groups: flowability, compressibility and packaging, stability and reconstitution, color properties, browning index, and foaming properties.

Milk type significantly influences flowability, compressibility, and packaging properties. For instance, skim milk powder, with its higher protein and lower fat content, exhibits greater density and compactness compared to whole milk powder. A comparable pattern was seen in the Carr Index (CI), while the Hausner Ratio (HR) remained consistent across samples, except for WOF powder, which exhibited poor flowability due to its high fat content.

The choice of evaporation and drying techniques primarily affects powder density. Powders produced via open-pan evaporation and spray drying tend to have denser, more spherical particles, resulting in improved packing density. In contrast, freeze-dried powders exhibit a porous structure with lower density. However, these treatments had minimal impact on overall flow properties, except for spray-dried WOS, which demonstrated better flowability due to its lower porosity and reduced particle adhesion caused by relative lower fat content

Water activity is influenced by milk type, evaporation method, and drying techniques. Spray drying and open-pan evaporation often yield powders with higher moisture content, which may compromise stability due to increased water activity. Drying methods also significantly impact hygroscopicity, which is linked to the presence of amorphous lactose and high protein content.

Solubility and wettability are primarily determined by milk type, with skim milk powder showing greater moisture absorption because of its higher protein and lactose content. Additionally, particle size plays a critical role, as smaller particles enhance these properties.

Milk type, evaporation, and drying methods also significantly influence the color parameters of milk powder, including Lightness (L^*), Green-Red (a^*), and Yellow-Blue (b^*). Higher processing temperatures result in darker samples, an increase in the a^* value (indicating reduced greenness), and a rise in the b^* value (indicating greater yellowness). Additionally, smaller particle size contributes to increased whiteness. The greenest samples are likely associated with the absence of fat and riboflavin absorption, while the yellowest samples are linked to fat content, which can retain carotenoids.

Although the type of milk, evaporation method, and drying technique did not significantly affect the Browning Index (BI), the study's samples exhibited higher BI values than those reported in the literature. This increased browning is likely due to prolonged heat exposure, particularly in spray drying (150°C) and open-pan evaporation (100.33°C), which accelerate Maillard reactions.

Foaming properties improve with higher protein content, as proteins enhance foam stability by forming elastic films around air bubbles. Conversely, a high fat content in whole milk powders can disrupt these films. Interactions between milk type, evaporator, and drying method significantly affect foam stability.

These findings underscore the importance of selecting appropriate milk types and processing methods to optimize the quality, functionality, and stability of milk powder for diverse applications. Additionally, this study provides valuable insights for the dairy industry, guiding the production of milk powder with consistent quality while minimizing costs. For instance, spray drying offers a cost-effective and efficient solution for large-scale production with desirable powder properties. On the other hand, freeze drying, though more expensive, is better suited for high-value bioactive products where preserving biofunctional properties is crucial.

Skimmed milk powder, with a higher protein and lower fat content, has a higher density and compactness than whole milk powder, but does not show significant differences in flow indices such as Hausner (HI) and Carr (CI). Powders produced by open pan evaporation and spray drying have denser and more spherical particles with better flowability, while freeze-dried powders have porous structures with lower density and quite reasonable flowability.

7. BIBLIOGRAPHY

- AL-HILPHY, A. R. et al. Assessing Compositional and Quality Parameters of Unconcentrated and Refractive Window Concentrated Milk Based on Color Components. **Dairy**, v. 3, n. 2, p. 400–412, 2022.
- ANEMA, S. G. **The whey proteins in milk: Thermal denaturation, physical interactions, and effects on the functional properties of milk**. 3. ed. [s.l.] Elsevier Inc., 2019.
- BABIO, N. et al. Consumption of Yogurt, Low-Fat Milk, and Other Low-Fat Dairy Products Is Associated with Lower Risk of Metabolic Syndrome Incidence in an Elderly Mediterranean Population1-3. **Journal of Nutrition**, v. 145, n. 10, p. 2308–2316, 2015.
- BALDELLI, A. et al. Spray freeze drying of dairy products: Effect of formulation on dispersibility. **Journal of Food Engineering**, v. 335, n. June, p. 111191, 2022.
- BARROS, M. V. et al. An analysis of Brazilian raw cow milk production systems and environmental product declarations of whole milk. **Journal of Cleaner Production**, v. 367, n. December 2021, 2022.
- BEZIE, A. The Effect of Different Heat Treatment on the Nutritional Value of Milk and Milk Products and Shelf-Life of Milk Products. A Review. **Journal of Dairy & Veterinary Sciences**, v. 11, n. 5, p. 1–8, 2019.
- BOJANA MILOVANOVIC, I. D. et al. What Is the Color of Milk and Dairy Products and. **Foods**, p. 1–17, 2020.
- BURGAIN, J. et al. Surface chemistry and microscopy of food powders. **Progress in Surface Science**, v. 92, n. 4, p. 409–429, 2017.
- CARPIN, M. et al. Caking of lactose: A critical review. **Trends in Food Science and Technology**, v. 53, p. 1–12, 2016.
- COŞKUN, N. et al. The Impact of Freeze Drying on Bioactivity and Physical Properties of Food Products. **Applied Sciences (Switzerland)**, v. 14, n. 20, 2024. **Applied Sciences (Switzerland)**, v. 14, n. 20, 2024.
- DE OLIVEIRA, A. H. et al. Influence of spray drying conditions on the properties of whole goat milk. **Drying Technology**, v. 39, n. 6, p. 726–737, 2021.
- DESHWAL, G. K. et al. Effect of spray and freeze drying on physico-chemical, functional, moisture sorption and morphological characteristics of camel milk powder. **Lwt**, v. 134, n. July, p. 110117, 2020.
- DING, H. et al. Effects of morphology on the bulk density of instant whole milk powder. **Foods**, v. 9, n. 8, p. 1–19, 2020.
- ERCAN, N.; TEL ADIGÜZEL, K. Effect of early childhood cow's milk elimination diet on eating behaviours, nutrition and growth status at age 2–6 years. **Journal of Human Nutrition and Dietetics**, v. 35, n. 2, p. 300–309, 2022.
- FARKYE, N. Y. Evaporated and Sweetened Condensed Milks. **Dairy Processing and Quality Assurance**, p. 309–318, 2019.
- FOURNAISE, T. et al. Impact of formulation on reconstitution and flowability of spray-dried milk powders. **Powder Technology**, v. 372, p. 107–116, 2020.

- GAIDHANI, K. A., HARWALKAR, M., BHAMBERE, D., & NIRGUDE, P. S. World Journal of Pharmaceutical research FORMULATION. **SJIF journal**, v. 2, n. 5, p. 1685–1703, 2021.
- GARCIA-AMEZQUITA, L. E. et al. Freeze-drying: The Basic Process. **Encyclopedia of Food and Health**, p. 104–109, 2015.
- GÓRSKA-WARSEWICZ, H. et al. Milk and dairy products and their nutritional contribution to the average polish diet. **Nutrients**, v. 11, n. 8, 2019.
- HAMBA, F. et al. Helicity transfer in rotary evaporator flow. **Physics of Fluids**, v. 26, n. 1, 2014.
- HAQUE, M. K.; ROOS, Y. H. Crystallization and X-ray diffraction of spray-dried and freeze-dried amorphous lactose. **Carbohydrate Research**, v. 340, n. 2, p. 293–301, 2005.
- HAYES, E. et al. Trend analysis and prediction of seasonal changes in milk composition from a pasture-based dairy research herd. **Journal of Dairy Science**, v. 106, n. 4, p. 2326–2337, 2023.
- HEMME, T. Dairy Report 2022: for a better understanding of the dairy world. **IFCN – International Farm Comparison Network**, 2022.
- HO, T. M. et al. Changes in physicochemical properties of spray-dried camel milk powder over accelerated storage. **Food Chemistry**, v. 295, p. 224–233, 2019.
- HO, T. M. et al. Foaming Properties and Foam Structure of Milk Determined by Its Protein Content and Protein to Fat Ratio. **Food and Bioprocess Technology**, n. 0123456789, 2024.
- HO, T. M.; BHANDARI, B.; BANSAL, N. **Influence of Milk Fat on Foam Formation, Foam Stability and Functionality of Aerated Dairy Products**. [s.l: s.n.]. v. 46
- HO, T. M.; BHANDARI, B. R.; BANSAL, N. Functionality of bovine milk proteins and other factors in foaming properties of milk: a review. **Critical Reviews in Food Science and Nutrition**, v. 62, n. 17, p. 4800–4820, 2022.
- HUPPERTZ, T.; GAZI, I. Lactose in dairy ingredients: Effect on processing and storage stability. **Journal of Dairy Science**, v. 99, n. 8, p. 6842–6851, 2016.
- IBRAHIM, A. H.; KHALIFA, S. A. Effect of freeze-drying on camel's milk nutritional properties. **International Food Research Journal**, v. 22, n. 4, p. 1438–1445, 2015.
- JAFARI, S. M.; GHALEGI GHALENOEI, M.; DEHNAD, D. Influence of spray drying on water solubility index, apparent density, and anthocyanin content of pomegranate juice powder. **Powder Technology**, v. 311, p. 59–65, 2017.
- JANGAM, S. V. An overview of recent developments and some R&D challenges related to drying of foods. **Drying Technology**, v. 29, n. 12, p. 1343–1357, 2011.
- Jl, J. et al. Rehydration behaviours of high protein dairy powders: The influence of agglomeration on wettability, dispersibility and solubility. **Food Hydrocolloids**, v. 58, p. 194–203, 2016.
- JUAREZ-ENRIQUEZ, E. et al. Effect of water content on the flowability of hygroscopic powders. **Journal of Food Engineering**, v. 205, p. 12–17, 2017.
- KALIT, S.; KALIT, M. T.; DOLEN, I. Fat and Protein Recovery , Yield and Sensory

- Properties of cki Škripavac Cheese. **Foods**, v. 10, 2021.
- KELLY, A.; O'CONNELL, J.; FOX, P. Manufacture and properties of Milk Powders. **Dairy Chemistry**, v. 2, p. 1027–1061, 2016.
- KERR, W. L. **Food drying and evaporation processing operations**. [s.l.] Elsevier Inc., 2019.
- KHOLER, M. A. Evaporation from Pans and Lakes. **Biologia Centrali-Americaa**, v. 2, p. v–413, 1955.
- LANGOVÁ, J.; ŠTENCL, J. Moisture sorption isotherms of whole milk powder in the temperature range of 5–35 °C and critical values of water activity prediction. **Acta Veterinaria Brno**, v. 83, p. S35–S40, 2014.
- LEE, J. et al. Unravelling the Influence of Composition and Heat Treatment on Key Characteristics of Dairy Protein Powders Using a Multifactorial Approach. **Foods**, v. 12, n. 17, 2023.
- LEE, K.; KIM, J. Dairy Food Consumption is Inversely Associated Korean Adults. **Nutrients**, v. 11, p. 1035, 2019.
- LIN, Y. et al. Effect of heat treatment, evaporation and spray drying during skim milk powder manufacture on the compositional and processing characteristics of reconstituted skim milk and concentrate. **International Dairy Journal**, v. 78, p. 53–64, 2018.
- MAHMOUD, M. S. Experimental Study to Evaluate Mist System Performance. **International Journal of Innovative Research in Advanced Engineering (IJIRAE)**, v. 2, n. 9, p. 41–48, 2014.
- MAIDANNYK, V. et al. Water sorption and hydration in spray-dried milk protein powders: Selected physicochemical properties. **Food Chemistry**, v. 304, p. 125418, 2020.
- MAURÍCIO, É. F. et al. Comparison of experimental setups for the production of milk concentrates and subsequent characterization. **Lwt**, v. 151, n. July, 2021.
- MEENA, G. S. et al. Physico-chemical , functional and rheological properties of milk protein concentrate 60 as affected by disodium phosphate addition , diafiltration and homogenization. **Journal of Food Science and Technology**, 2017.
- MICHALSKA, A. et al. The influence of inulin on the retention of polyphenolic compounds during the drying of blackcurrant juice. **Molecules**, v. 24, n. 22, 2019.
- MILOVANOVIC, B. et al. What is the color of milk and dairy products and how is it measured? **Foods**, v. 9, n. 11, p. 1–17, 2020.
- MOHAN, M. S. et al. Milk fat : opportunities , challenges and innovation Milk fat : opportunities , challenges and innovation. **Critical Reviews in Food Science and Nutrition**, v. 0, n. 0, p. 1–33, 2020.
- NASSER, S. et al. Influence of storage conditions on the functional properties of micellar casein powder. **Food and Bioproducts Processing**, v. 106, p. 181–192, 2017.
- OAKLEY, D. E. Spray dryer modeling in theory and practice. **Drying Technology**, v. 22, n. 6, p. 1371–1402, 2004.
- PONNAL, R. P. et al. Colorimetry of dairy products. **International Dairy Journal**, v. 113, p.

104886, 2021.

PUGLIESE, A. et al. Physical characterization of whole and skim dried milk powders. **Journal of Food Science and Technology**, v. 54, n. 11, p. 3433–3442, 2017.

REGULAMENTOS TÉCNICOS DE IDENTIDADE E QUALIDADE (RTIQ). **Regulamento Técnico De Identidade E Qualidade De Apresentado**. , 2018.

ROMO, M. et al. Pulse Spray Drying for Bovine Skimmed Milk Powder Production. **Foods**, v. 13, n. 6, 2024.

ROSA, E.; PRUDENCIO, E. S. A comprehensive approach about comparison between drying technologies and powdered dairy products. **Food Research International**, v. 173, n. P1, p. 113326, 2023.

ROY, I.; BHUSHANI, A.; ANANDHARAMAKRISHNAN, C. Techniques for the Preconcentration of Milk. **Handbook of Drying for Dairy Products**, p. 23–41, 2017.

RUPP, L. S.; MOLITOR, M. S.; LUCEY, J. A. Effect of processing methods and protein content of the concentrate on the properties of milk protein concentrate with 80% protein. **Journal of Dairy Science**, v. 101, n. 9, p. 7702–7713, 2018.

RYABOVA, A. E.; SEMIPYATNY, V. K.; GALSTYAN, A. G. Effects of storage conditions on milk powder properties. **Journal of Dairy Science**, v. 106, n. 10, p. 6741–6758, 2023.

RYAN, G.; O'REGAN, J.; FITZGERALD, R. J. Foaming and sensory properties of bovine milk protein isolate and its associated enzymatic hydrolysates. **International Dairy Journal**, v. 137, p. 105511, 2023.

SAHA, D.; NANDA, S. K.; YADAV, D. N. Optimization of spray drying process parameters for production of groundnut milk powder. **Powder Technology**, v. 355, p. 417–424, 2019.

SAJAL, M. The Effect of Dryer Load on Freeze Drying Process Design. **Journal of pharmaceutical sciences**, v. 101, n. 7, p. 2271–2280, 2012.

SAMSU, Z. A.; ZAHIRAH, A.; ZAHIR, M. Production of oil palm milk powder by spray drying technique *Materials Today : Proceedings* Production of oil palm milk powder by spray drying technique. **Materials Today: Proceedings**, v. 31, n. October, p. 306–312, 2020.

SCHIANO, A. N.; HARWOOD, W. S.; DRAKE, M. A. A 100-Year Review: Sensory analysis of milk. **Journal of Dairy Science**, v. 100, n. 12, p. 9966–9986, 2017.

SCHUCK, P. et al. Recent advances in spray drying relevant to the dairy industry: A comprehensive critical review. **Drying Technology**, v. 34, n. 15, p. 1773–1790, 2016.

SEO, C. W. Improved Flowability and Wettability of Whey Protein-Fortified Skim Milk Powder via Fluidized Bed Agglomeration. **Food Science of Animal Resources**, v. 42, n. 6, p. 915–927, 2022.

SHILPASHREE, B. G. et al. LWT - Food Science and Technology Succinylation of sodium caseinate and its effect on physicochemical and functional properties of protein. **LWT - Food Science and Technology**, v. 64, n. 2, p. 1270–1277, 2015.

SILVA, J. V. C.; O'MAHONY, J. A. Flowability and wetting behaviour of milk protein ingredients as influenced by powder composition, particle size and microstructure. **International Journal of Dairy Technology**, v. 70, n. 2, p. 277–286, 2017.

- SIVAKUMAR, R. et al. Fluidized bed drying of some agro products – A review. **Renewable and Sustainable Energy Reviews**, v. 61, p. 280–301, 2016.
- SMITH, N. W. et al. Modeling the Contribution of Milk to Global Nutrition. **Frontiers in Nutrition**, v. 8, n. January, p. 1–7, 2022.
- SUDHARSHAN, R. et al. Influence of processing conditions on functional and reconstitution properties of milk powder made from Osmanabadi goat milk by spray drying. **Small Ruminant Research**, v. 119, n. 1–3, p. 130–137, 2014.
- SUHAG, R.; KELLIL, A.; RAZEM, M. Factors Influencing Food Powder Flowability. **Powders**, v. 3, n. 1, p. 65–76, 2024.
- SULIEMAN, A. M. E. et al. Comparison of Physicochemical Properties of Spray-dried Camel 's Milk and Cow 's Milk Powder. **International Journal of Food Science and Nutrition Engineering**, v. 4, n. October, p. 3–8, 2015.
- TASTEMIROVA, U.; CIPROVICA, I.; SHINGISOV, A. The comparison of the spray-drying and freeze-drying techniques for camel milk: A review. **Research for Rural Development**, v. 35, p. 102–105, 2020.
- TERESA, T. D. et al. Self-Evaluation of Gambella University. **International Journal of Research -GRANTHAALAYAH**, v. 6, n. 5, p. 201–228, 2018.
- UEDA, J. M. et al. Powdered Foods: Structure, Processing, and Challenges: A Review. **Applied Sciences (Switzerland)**, v. 13, n. 22, 2023.
- WANG, B. et al. Characteristics of bovine lactoferrin powders produced through spray and freeze drying processes. **International Journal of Biological Macromolecules**, v. 95, p. 985–994, 2017.
- WANG, Y. et al. Remineralization of early enamel caries lesions using different bioactive elements containing toothpastes: An in vitro study. **Technology and Health Care**, v. 24, n. 5, p. 701–711, 2016.
- WHITEPAPER, R. E. Concentration of Dairy Permeate for Lactose Crystallization. **Heidolph**, v. 1, n. April, 2021.
- WOO, M. W.; BHANDARI, B. Spray drying for food powder production. **Handbook of Food Powders: Chemistry and Technology, Second Edition**, p. 19–36, 2023.
- ZAFISAH, N. S. et al. Processing of raw coconut milk for its value addition using spray and freeze drying techniques. **Journal of Food Process Engineering**, v. 41, n. 1, p. 1–11, 2018.
- ZHOU, Z.; LANGRISH, T. Color formation and Maillard reactions during the spray drying process of skim milk and model systems. **Journal of Food Process Engineering**, v. 45, n. 1, 2022.
- ZOUARI, A. et al. Changes in physical and biochemical properties of spray dried camel and bovine milk powders. **Lwt**, v. 128, p. 109437, 2020a.
- ZOUARI, A. et al. Changes in physical and biochemical properties of spray dried camel and bovine milk powders. **Lwt**, v. 128, n. April, p. 109437, 2020b.
- ZOUARI, A. et al. Effect of spray-drying parameters on the solubility and the bulk density of camel milk powder: A response surface methodology approach. **International Journal of Dairy Technology**, v. 73, n. 3, p. 616–624, 2020c.

ANNEXE 1.

Tables 1.A, 1.B, 1.C, 1.D, and 1.E show the analysis of variance (ANOVA) for chemical composition as a function of milk type, concentration, and drying technique.

Table 1.A-Analysis of Variance (ANOVA) for Protein Content

| | Coeff. | Effect | Std.Err. | t(17) | p |
|------------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 26,25369 | 26,25369 | 0,365677 | 71,79466 | 0,000000 |
| (1)Type of milk | 3,05237 | 6,10474 | 0,731355 | 8,34717 | 0,000000 |
| (2)Evaporation | 0,36857 | 0,73713 | 0,731355 | 1,00790 | 0,327633 |
| (3)Dryer | -0,41296 | -0,82592 | 0,731355 | -1,12930 | 0,274454 |
| 1 by 2 | 0,22167 | 0,44333 | 0,731355 | 0,60618 | 0,552406 |
| 1 by 3 | 0,16425 | 0,32850 | 0,731355 | 0,44916 | 0,658989 |
| 2 by 3 | -0,29624 | -0,59247 | 0,731355 | -0,81010 | 0,429078 |

Table 1.B-Analysis of Variance (ANOVA) for Fat Content

| | Coeff. | Effect | Std.Err. | t(17) | p |
|------------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 11,76250 | 11,7625 | 0,183778 | 64,0037 | 0,000000 |
| (1)Type of milk | -5,92500 | -11,8500 | 0,367557 | -32,2399 | 0,000000 |
| (2)Evaporation | 0,88750 | 1,7750 | 0,367557 | 4,8292 | 0,000157 |
| (3)Dryer | 0,63750 | 1,2750 | 0,367557 | 3,4689 | 0,002936 |
| 1 by 2 | -0,30000 | -0,6000 | 0,367557 | -1,6324 | 0,120979 |
| 1 by 3 | -0,55000 | -1,1000 | 0,367557 | -2,9927 | 0,008180 |
| 2 by 3 | -1,11250 | -2,2250 | 0,367557 | -6,0535 | 0,000013 |

Table 1.C-Analysis of Variance (ANOVA) for Moisture Content

| | Coeff. | Effect | Std.Err. | t(17) | p |
|------------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 3,664583 | 3,664583 | 0,039834 | 91,99558 | 0,000000 |
| (1)Type of milk | -0,384583 | -0,769167 | 0,079669 | -9,65457 | 0,000000 |
| (2)Evaporation | -0,093750 | -0,187500 | 0,079669 | -2,35350 | 0,030892 |
| (3)Dryer | -0,192917 | -0,385833 | 0,079669 | -4,84297 | 0,000152 |
| 1 by 2 | -0,051250 | -0,102500 | 0,079669 | -1,28658 | 0,215487 |
| 1 by 3 | 0,257917 | 0,515833 | 0,079669 | 6,47473 | 0,000006 |
| 2 by 3 | 0,060417 | 0,120833 | 0,079669 | 1,51670 | 0,147718 |

Table 1.D-Analysis of Variance (ANOVA) for Ash Content

| | Coeff. | Effect | Std.Err. | t(16) | p |
|------------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 14,83884 | 14,83884 | 0,231151 | 64,19555 | 0,000000 |
| (1)Type of milk | -0,89835 | -1,79670 | 0,462301 | -3,88643 | 0,001311 |
| (2)Evaporation | -0,11300 | -0,22600 | 0,462301 | -0,48886 | 0,631571 |
| (3)Dryer | 0,47835 | 0,95670 | 0,462301 | 2,06942 | 0,055056 |
| 1 by 2 | -0,82538 | -1,65077 | 0,462301 | -3,57076 | 0,002552 |
| 1 by 3 | -0,77274 | -1,54549 | 0,462301 | -3,34303 | 0,004127 |
| 2 by 3 | 1,52138 | 3,04275 | 0,462301 | 6,58176 | 0,000006 |

Table 1.E-Analysis of Variance (ANOVA) for Carbohydrate Content

| | Coeff. | Effect | Std.Err. | t(16) | p |
|------------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 43,48039 | 43,48039 | 0,433041 | 100,4071 | 0,000000 |
| (1)Type of milk | 4,15556 | 8,31113 | 0,866082 | 9,5962 | 0,000000 |
| (2)Evaporation | -1,04932 | -2,09863 | 0,866082 | -2,4231 | 0,027616 |
| (3)Dryer | -0,50997 | -1,01994 | 0,866082 | -1,1776 | 0,256157 |
| 1 by 2 | 0,95497 | 1,90994 | 0,866082 | 2,2053 | 0,042412 |
| 1 by 3 | 0,90058 | 1,80116 | 0,866082 | 2,0797 | 0,053994 |
| 2 by 3 | -0,17306 | -0,34612 | 0,866082 | -0,3996 | 0,694709 |

ANNEXE 2.

Tables 2.A, 2.B, 2.C, and 2.D show the analysis of variance (ANOVA) for flowability, compressibility, and packing properties: bulk density, compact density, Carr's Index (CI) and Hausner Ratio (HR) as a function of milk type, concentration, and drying technique.

Table 2.A-Analysis of Variance (ANOVA) for Bulk Density

| | Effect | Coeff. | Std.Err. | t(17) | p |
|-----------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 0,373633 | 0,373633 | 0,006394 | 58,43946 | 0,000000 |
| (1)T milk | 0,060596 | 0,030298 | 0,012787 | 4,73886 | 0,000190 |
| (2)Evaporation | -0,015051 | -0,007525 | 0,012787 | -1,17704 | 0,255397 |
| (3)Dryer | -0,054498 | -0,027249 | 0,012787 | -4,26194 | 0,000526 |
| 1 by 2 | 0,006473 | 0,003236 | 0,012787 | 0,50618 | 0,619233 |
| 1 by 3 | 0,030483 | 0,015241 | 0,012787 | 2,38386 | 0,029059 |
| 2 by 3 | 0,057583 | 0,028791 | 0,012787 | 4,50320 | 0,000314 |

Table 2.B-Analysis of Variance (ANOVA) for Compact Density

| | Effect | Coeff. | Std.Err. | t(17) | p |
|------------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 0,479063 | 0,479063 | 0,003621 | 132,3157 | 0,000000 |
| (1)Type of milk | 0,098623 | 0,049312 | 0,007241 | 13,6197 | 0,000000 |
| (2)Evaporation | -0,023218 | -0,011609 | 0,007241 | -3,2064 | 0,005176 |
| (3)Dryer | -0,059357 | -0,029678 | 0,007241 | -8,1971 | 0,000000 |
| 1 by 2 | 0,022707 | 0,011353 | 0,007241 | 3,1358 | 0,006024 |
| 1 by 3 | 0,006755 | 0,003377 | 0,007241 | 0,9329 | 0,363955 |
| 2 by 3 | 0,024380 | 0,012190 | 0,007241 | 3,3668 | 0,003661 |

Table 2.C-Analysis of Variance (ANOVA) for Carr's Index (CI)

| | Coeff. | Effect | Std.Err. | t(17) | p |
|-----------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 20,02780 | 20,02780 | 0,530812 | 37,73048 | 0,000000 |
| (1)T milk | 2,42824 | 4,85648 | 1,061624 | 4,57458 | 0,000269 |
| (2)Evaporation | -0,99181 | -1,98363 | 1,061624 | -1,86848 | 0,079028 |
| (3)Dryer | -0,00930 | -0,01860 | 1,061624 | -0,01752 | 0,986225 |
| 1 by 2 | 0,53625 | 1,07250 | 1,061624 | 1,01024 | 0,326544 |
| 1 by 3 | -2,63474 | -5,26948 | 1,061624 | -4,96360 | 0,000118 |
| 2 by 3 | -2,02483 | -4,04967 | 1,061624 | -3,81459 | 0,001386 |

Table 2.D-Analysis of Variance (ANOVA) for Hausner Ratio (HR)

| | Coeff. | Effect | Std.Err. Pure Err | t(16) | p |
|-----------------------|---------------|---------------|------------------------------|--------------|-----------------|
| Mean/Interc. | 1,297974 | 1,297974 | 0,028802 | 45,06619 | 0,000000 |
| (1)T milk | 0,012630 | 0,025259 | 0,057603 | 0,43851 | 0,666880 |
| (2)Evaporation | -0,022985 | -0,045970 | 0,057603 | -0,79805 | 0,436528 |
| (3)Dryer | 0,022950 | 0,045901 | 0,057603 | 0,79685 | 0,437204 |
| 1 by 2 | 0,036754 | 0,073508 | 0,057603 | 1,27612 | 0,220126 |
| 1 by 3 | -0,051551 | -0,103101 | 0,057603 | -1,78986 | 0,092418 |
| 2 by 3 | -0,077368 | -0,154735 | 0,057603 | -2,68624 | 0,016223 |

ANNEXE 3.

Tables 3.A, 3.B, 3.C, and 3.D show the analysis of variance (ANOVA) for stability and reconstitution properties: water activity, hygroscopicity, solubility and wettability as a function of milk type, concentration, and drying technique.

Table 3.A-Analysis of Variance (ANOVA) for Water Activity.

| | Effect | Coeff. | Std.Err. | t(16) | p |
|-----------------------|-----------|-----------|----------|----------|----------|
| Mean/Interc. | 0,229917 | 0,229917 | 0,001615 | 142,3319 | 0,000000 |
| (1)T milk | -0,041500 | -0,020750 | 0,003231 | -12,8455 | 0,000000 |
| (2)Evaporation | -0,097500 | -0,048750 | 0,003231 | -30,1791 | 0,000000 |
| (3)Dryer | -0,160000 | -0,080000 | 0,003231 | -49,5247 | 0,000000 |
| 1 by 2 | -0,041500 | -0,020750 | 0,003231 | -12,8455 | 0,000000 |
| 1 by 3 | 0,026667 | 0,013333 | 0,003231 | 8,2541 | 0,000000 |
| 2 by 3 | 0,062000 | 0,031000 | 0,003231 | 19,1908 | 0,000000 |

Table 3.B-Analysis of Variance (ANOVA) for Hygroscopicity.

| | Coeff. | Effect | Std.Err. | t(17) | p |
|-----------------------|-----------|-----------|----------|----------|----------|
| Mean/Interc. | 9,571631 | 9,571631 | 0,247525 | 38,66937 | 0,000000 |
| (1)T milk | 0,065044 | 0,130088 | 0,495050 | 0,26278 | 0,795881 |
| (2)Evaporation | -0,191892 | -0,383784 | 0,495050 | -0,77524 | 0,448846 |
| (3)Dryer | 0,605696 | 1,211392 | 0,495050 | 2,44701 | 0,025568 |
| 1 by 2 | -0,046873 | -0,093746 | 0,495050 | -0,18937 | 0,852047 |
| 1 by 3 | -0,003626 | -0,007253 | 0,495050 | -0,01465 | 0,988481 |
| 2 by 3 | -0,215189 | -0,430377 | 0,495050 | -0,86936 | 0,396758 |

Table 3.C-Analysis of Variance (ANOVA) for Solubility.

| | Coeff. | Effect | Std.Err. | t(17) | p |
|-----------------------|----------|----------|----------|----------|----------|
| Mean/Interc. | 74,73200 | 74,73200 | 0,781304 | 95,65031 | 0,000000 |
| (1)T milk | 9,82952 | 19,65904 | 1,562609 | 12,58091 | 0,000000 |
| (2)Evaporation | 0,39717 | 0,79434 | 1,562609 | 0,50834 | 0,617748 |
| (3)Dryer | -0,70527 | -1,41055 | 1,562609 | -0,90269 | 0,379304 |
| 1 by 2 | 2,40796 | 4,81592 | 1,562609 | 3,08197 | 0,006761 |
| 1 by 3 | -3,58103 | -7,16207 | 1,562609 | -4,58340 | 0,000264 |
| 2 by 3 | -1,88323 | -3,76646 | 1,562609 | -2,41037 | 0,027542 |

Table 3.D-Analysis of Variance (ANOVA) for Wettability.

| | Effect | Std.Err. | t(17) | Coeff. | p |
|-----------------------|---------------|-----------------|--------------|---------------|-----------------|
| Mean/Interc. | 66,222 | 8,74615 | 7,57154 | 66,2219 | 0,000001 |
| (1)T milk | -117,803 | 17,49231 | -6,73455 | -58,9014 | 0,000003 |
| (2)Evaporation | -18,102 | 17,49231 | -1,03487 | -9,0512 | 0,315225 |
| (3)Dryer | 15,696 | 17,49231 | 0,89729 | 7,8478 | 0,382098 |
| 1 by 2 | 21,959 | 17,49231 | 1,25535 | 10,9795 | 0,226332 |
| 1 by 3 | -11,590 | 17,49231 | -0,66260 | -5,7952 | 0,516468 |
| 2 by 3 | -73,138 | 17,49231 | -4,18116 | -36,5691 | 0,000627 |

ANNEXE 4.

Tables 4.A, 4.B, 4.C, and 4.D show the analysis of variance (ANOVA) for the color properties: L*, a*, b* and browning Index (BI) as a function of milk type, concentration, and drying technique.

Table 4.A-Analysis of Variance (ANOVA) for L*.

| | Effect | Coeff. | Std.Err. | t(17) | p |
|-----------------|----------|----------|----------|----------|----------|
| Mean/Interc. | 91,81417 | 91,81417 | 0,097893 | 937,9029 | 0,000000 |
| (1)Type of milk | 0,63500 | 0,31750 | 0,195786 | 3,2433 | 0,004780 |
| (2)Evaporation | 0,40667 | 0,20333 | 0,195786 | 2,0771 | 0,043265 |
| (3)Dryer | -0,86000 | -0,43000 | 0,195786 | -4,3925 | 0,000397 |
| 1 by 2 | 0,42333 | 0,21167 | 0,195786 | 2,1622 | 0,045149 |
| 1 by 3 | 0,47000 | 0,23500 | 0,195786 | 2,4006 | 0,028094 |
| 2 by 3 | -0,95833 | -0,47917 | 0,195786 | -4,8948 | 0,000137 |

Table 4.B-Analysis of Variance (ANOVA) for a*.

| | Effect | Std.Err. | t(17) | Coeff. | p |
|-----------------|----------|----------|----------|-----------|----------|
| Mean/Interc. | -0,74458 | 0,024749 | -30,0855 | -0,744583 | 0,000000 |
| (1)Type of milk | -0,74917 | 0,049498 | -15,1353 | -0,374583 | 0,000000 |
| (2)Evaporation | -1,01917 | 0,049498 | -20,5901 | -0,509583 | 0,000000 |
| (3)Dryer | 0,44583 | 0,049498 | 9,0071 | 0,222917 | 0,000000 |
| 1 by 2 | -0,68250 | 0,049498 | -13,7885 | -0,341250 | 0,000000 |
| 1 by 3 | 0,00250 | 0,049498 | 0,0505 | 0,001250 | 0,960307 |
| 2 by 3 | 0,29583 | 0,049498 | 5,9767 | 0,147917 | 0,000015 |

Table 4.C-Analysis of Variance (ANOVA) for b*.

| | Effect | Std.Err. | t(17) | Coeff. | p |
|----------------|----------|----------|----------|----------|----------|
| Mean/Interc. | 20,50208 | 0,396413 | 51,71904 | 20,50208 | 0,000000 |
| (1)T milk | 0,14250 | 0,792825 | 0,17974 | 0,07125 | 0,859484 |
| (2)Evaporation | -2,48917 | 0,792825 | -3,13962 | -1,24458 | 0,005975 |
| (3)Dryer | 1,98583 | 0,792825 | 2,50476 | 0,99292 | 0,022725 |
| 1 by 2 | 2,18250 | 0,792825 | 2,75281 | 1,09125 | 0,013588 |
| 1 by 3 | -1,68250 | 0,792825 | -2,12216 | -0,84125 | 0,048817 |
| 2 by 3 | 2,88250 | 0,792825 | 3,63573 | 1,44125 | 0,002044 |

Table 4.D-Analysis of Variance (ANOVA) for BI.

| | Coeff. | Effect | Std.Err. | t(17) | p |
|-----------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 25,95165 | 25,95165 | 1,087179 | 23,87062 | 0,000000 |
| (1)T milk | 0,50647 | 1,01294 | 2,174359 | 0,46586 | 0,647231 |
| (2)Evaporation | -0,20548 | -0,41095 | 2,174359 | -0,18900 | 0,852332 |
| (3)Dryer | -0,68316 | -1,36632 | 2,174359 | -0,62838 | 0,538108 |
| 1 by 2 | -0,53581 | -1,07162 | 2,174359 | -0,49284 | 0,628428 |
| 1 by 3 | -0,89615 | -1,79229 | 2,174359 | -0,82429 | 0,421193 |
| 2 by 3 | -1,40904 | -2,81808 | 2,174359 | -1,29605 | 0,212278 |

ANNEXE 5.

Tables 5.A and 5.B show the analysis of variance (ANOVA) for the color properties Functional Properties: foam capacity and stability as a function of milk type, concentration, and drying technique.

Table 5.A-Analysis of Variance (ANOVA) for Foam Capacity

| | Effect | Coeff. | Std.Err. | t(17) | p |
|------------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 102,9922 | 102,9922 | 0,172371 | 597,5044 | 0,000000 |
| (1)Type of milk | -2,3973 | -1,1987 | 0,344741 | -6,9540 | 0,000002 |
| (2)Evaporation | 0,1398 | 0,0699 | 0,344741 | 0,4056 | 0,690109 |
| (3)Dryer | 0,2488 | 0,1244 | 0,344741 | 0,7217 | 0,480314 |
| 1 by 2 | -0,4260 | -0,2130 | 0,344741 | -1,2358 | 0,233320 |
| 1 by 3 | 0,0451 | 0,0225 | 0,344741 | 0,1307 | 0,897547 |
| 2 by 3 | 0,6644 | 0,3322 | 0,344741 | 1,9272 | 0,070840 |

Table 5.B-Analysis of Variance (ANOVA) for Foam Stability

| | Effect | Coeff. | Std.Err. | t(17) | p |
|------------------------|---------------|---------------|-----------------|--------------|-----------------|
| Mean/Interc. | 99,89581 | 99,89581 | 0,140535 | 710,8270 | 0,000000 |
| (1)Type of milk | 0,41127 | 0,20563 | 0,281069 | 1,4632 | 0,161652 |
| (2)Evaporation | 0,21602 | 0,10801 | 0,281069 | 0,7686 | 0,452704 |
| (3)Dryer | 0,58871 | 0,29436 | 0,281069 | 2,0946 | 0,051500 |
| 1 by 2 | 0,12264 | 0,06132 | 0,281069 | 0,4363 | 0,668091 |
| 1 by 3 | 1,03297 | 0,51649 | 0,281069 | 3,6751 | 0,001876 |
| 2 by 3 | -1,12262 | -0,56131 | 0,281069 | -3,9941 | 0,000939 |