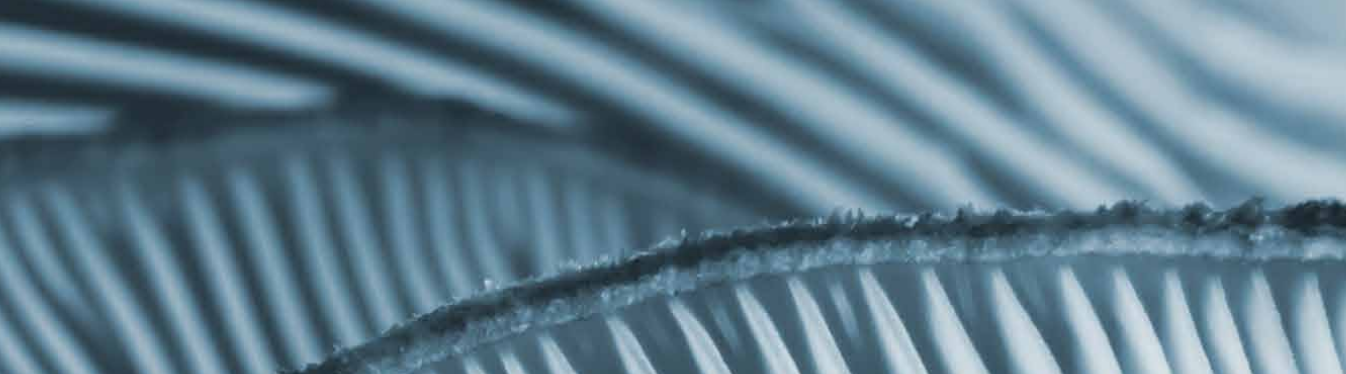




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Novel Drying Technologies in Food Science

Edited by Jayani Chandrapala



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Aims and Scope of the Series

The significance of food is undeniable, especially in light of the impending challenge facing humanity: ensuring there will be enough food to meet the basic needs of a population expected to reach approximately 10 billion by 2050. These food-related challenges align with some of the United Nations' sustainable development goals, with a target to achieve them by 2030. One thing is certain: food should be not only nourishing and safe but also tailored to the diverse needs of individuals throughout their lifetimes, all while meeting consumers' sensory expectations. Understanding the diverse chemical composition of food, often referred to as biodiversity, and how these components can contribute to human health by considering factors like bioaccessibility, bioavailability, and bioactivity at the organ level, is crucial for grasping and promoting a healthy diet. Thanks to the continuous evolution of analytical methods and interdisciplinary research, significant strides have been made in the field of food science and nutrition.

Meet the Series Editor



Maria Rosário Bronze has been working in Analytical Chemistry since 1986. Her Ph.D. in 1999 contributed to the study of food products using capillary electrophoresis. The main goal of her research since 1999 has been focused on Analytical Chemistry applied mainly to the analysis of foods and by-products of food industry. She conducted research in collaboration with national and international research groups, at iBET and ITQB Technology Division. From 2017 until 2021 she was head of Food & Health Division at iBET and head of the Food Functionality and Bioactives Laboratory. MR Bronze has been an Associate Professor at the Pharmacy Faculty of Lisbon University and head of the Structural Analysis Laboratory since 2012. As a researcher, MR Bronze is a Senior Scientific Advisor at Food & Health Division at iBET and Head of Food Functionality and Bioactives Laboratory at the same Institute, Collaborator at iMED and Researcher at ITQB NOVA. Her current research is focused on quality and beneficial health effects of food components. Gas and liquid chromatography associated with mass spectrometry are used by MR Bronze in the characterization of samples. Sensory evaluation is also an important area of her research. The main food products studied by her are olive tree products (olive, olive oil, leaves), cereals such as maize, legumes (faba bean, pea, chickpea, lentils) fruits (apple, grapes, opuntia ficus), fruit juices and wine, among others. More recently her interests have also involved biodiversity, bioaccessibility, and bioavailability studies on food products and their components, mainly phytochemicals as phenolic compounds, using different analytical tools such as mass spectrometry. As a senior scientific advisor at Food & Health Division at iBET she is involved in different areas: (i) isolation, characterization and formulation of bioactive and functional compounds or extracts from natural sources and wastes from food and other related industries; (ii) pre-clinical assays to provide support to understand health claims related with the beneficial effects of food nutrients/bioactive components; (iii) establishment of analytical methodologies including mass spectrometry state-of-the-art to fully characterize different matrices, from food products, natural extracts or biological fluids (Food Functionality and Bioactives Laboratory).

Meet the Volume Editor



Dr. Jayani Chandrapala is a globally recognized researcher and academic leader specializing in food chemistry and dairy science. She is the Head of the Department of Food Technology and Nutrition at RMIT University, Australia. Her research expertise includes the physical chemistry of dairy foods, protein conformations, and the interactions among food components, including protein-protein, protein-sugar, and protein-mineral dynamics. She is also renowned for her work in advanced membrane processing for waste valorization, as well as in emerging thermal and non-thermal technologies, and in the development of value-added and functional food products, including emulsions, microencapsulation, cheese, and yoghurt. She has over 180 publications, an H-index of 43, and serves on the editorial boards of several leading food science journals.

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Preface

The field of food drying and dehydration has undergone remarkable changes in recent decades, driven by the growing demand for sustainable, nutritious, and shelf-stable food products. As the global population continues to rise and food systems face increasing challenges related to sustainability, efficiency, and nutrition preservation, advanced drying technologies are playing an increasingly critical role in food processing and preservation.

This book, *Novel Drying Technologies in Food Science*, brings together diverse perspectives from researchers working across key areas of drying science and technology. It provides insight into both traditional and emerging techniques, ranging from conventional spray drying to cutting-edge approaches such as nanoencapsulation and infrared drying.

The chapters presented here explore not only the fundamental concepts and mechanisms of various drying techniques but also their real-world applications and the latest advancements that align with sustainable food production. Topics such as spray drying for food processing, nanoencapsulation for bioactive delivery, optimisation of infrared drying, and preservation strategies for nutritionally significant crops, such as purple mashua, highlight the diversity and relevance of research in this field.

We hope this book will serve as a valuable resource for researchers, students, food technologists, and industry professionals engaged in improving food preservation while maintaining quality, nutritional value, and environmental responsibility.

We extend our sincere gratitude to all the contributing authors for sharing their expertise and to the editorial and review teams whose support made this publication possible.

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Introductory Chapter: Novel Drying Technologies in Food Science

Jayani Chandrapala

1. Introduction

Drying is one of the oldest and most widely applied methods of food preservation. It facilitates storage and transport of food ingredients and products by reducing their weight and volume, while at the same time inhibiting microbial growth and extending shelf life [1]. Furthermore, drying contributes to nutritional security by making foods available year-round and reducing postharvest losses [2]. In this way, drying plays an important role in economic, nutritional, and sustainability contexts, supporting food security, reducing waste, and ensuring a constant supply of food for a growing population with limited resources [3].

Conventional methods such as sun, hot air and vacuum drying are widely used due to their simplicity, low cost, and ease of operation, but result in longer processing times and degradation of quality such as changes in flavour, texture, colour, and nutritional value [3]. Nowadays, consumers increasingly prefer clean-label, minimally processed, and nutrient-rich foods, creating a pressing need for innovative drying technologies that preserve sensitive nutrients while using energy more efficiently. Recent advances in novel drying technologies such as ultrasound, microwave, infrared, radio frequency, and their hybrid systems have shown significant improvements in drying efficiency, energy consumption, retention of nutrients and keeping the sensory attributes [4]. These novel techniques enhance the product quality while addressing sustainability challenges, making them highly promising for industrial applications. On the contrary, the drying efficiency and its influence on the physical, chemical, structural and functional characterisations of various food systems depend on the drying technology, their processing variables and food matrix and composition. Thus, future research should focus on optimising process parameters, scalability, and cost-effectiveness.

This book explores some of these novel drying technologies in depth, examining their underlying principles, process efficiencies, and food applications. Conventional and advanced methods will be compared while highlighting both opportunities and challenges in shaping the next generation of drying technologies that can better support industry needs. Simultaneously, these novel techniques will achieve global goals for food security, sustainability, and consumer health.

2. Traditional and novel drying technologies

Food drying is a complex process involving simultaneous heat and mass transfer, where water is removed from food materials [5]. Techniques such as freeze-drying, vacuum drying, osmotic dehydration, microwave-assisted, infrared, ultrasonic, electrohydrodynamic, and spray drying each offer distinct mechanisms and advantages. These techniques are often applied to high-value and heat-sensitive foods. Sometimes, these techniques are applied in combination to minimise further thermal damage and improve the efficiency. However, the drying outcomes are influenced by variables such as temperature, airflow, product type, thickness, and moisture content, which in turn affect drying time, energy use, and final product characteristics. Optimisation of the processing parameters possess many challenges due to the relationship of product properties, drying parameters, and equipment. Thus, a comprehensive understanding of drying principles and practical constraints is needed.

For instance, microwave drying (MD) uses volumetric heating, where electromagnetic waves interact with water molecules, allowing rapid inside-out drying. MD reduces drying time, improves energy efficiency, and retains bioactive compounds such as phenolics, vitamin C, and antioxidants [6]. However, uneven heating may cause hot spots, scorching, and instability of additives in the food system. Hybrid MD methods such as microwave-vacuum, fluidised bed, infrared, and freeze-drying may address these drawbacks, improving the product quality, functionality, and nutrient retention. But cost may be an unfavourable factor [7] which then restricts the large-scale adoption under commercial scale. Another technique is radio frequency drying (RFD), which operates at 1–300 MHz, producing internal dielectric heating for deeper penetration, uniform temperature distribution, and better nutrient retention. RFD permits rapid drying. However, optimisation of conditions is needed to balance nutrient stability and structural changes within a food system. Hybrid approaches such as RF-hot air or RF-vacuum significantly reduce drying time, energy use, and improve colour, antioxidants, and microstructure [8]. However, non-uniform heating and moisture distribution remain as the main challenges based on the nature of the food matrix [9]. Infrared (IR) drying which uses electromagnetic waves (0.75–1000 μm), transfer energy directly into food, allowing a high thermal efficiency, faster heating and good nutrient and colour retention. The wavelength plays a significant role in determining the efficacy of this technique across different food matrices [7], while drying performance depends on factors such as IR power, intensity, emitter distance, and slice thickness, with higher power generally reducing drying time but risking nutrient degradation. Hybrid IR techniques such as IR-freeze-drying, IR-vacuum, and IR-hot air have shown more benefits including faster drying, improved rehydration, higher retention of phenolics, vitamin C, and antioxidants, and lower energy use [10]. But, future work should more focus on optimising IR parameters, use of advance modelling methods for kinetics and moisture distribution, and scaling up in industrial applications [11]. Ultrasound is another promising non-thermal, energy-efficient method that accelerates drying by enhancing mass and heat transfer through acoustic cavitation, sponge effects, and mechanical vibrations. US helps to preserve colour, texture, and bioactive compounds, while the effects are majorly influenced by intensity, frequency, and transducer type *via* direct-contact or contactless modes. Similar to other techniques, industrial use remains limited mainly due to equipment costs [12]. These novel techniques are widely applied and compared with conventional drying within fruits, vegetables, dairy, meat, and grains to reduce

postharvest losses, extend shelf life, retain nutrients, improve stability and functionality, and increase quality [13], although each technique provides pros and cons of their own which limits scale-up applications.

3. Conclusion and future perspectives

Novel drying technologies include infrared drying, microwave drying, ultrasound-assisted drying, radio frequency drying, and their hybrid combinations. These technologies have shown great promise in overcoming the limitations of conventional drying methods including long drying times, high energy usages, and nutritional, structural, and sensory losses within variety of foods. For fruits, vegetables, and grains, the use of these advanced methods not only enhances product quality but also contributes to sustainability by minimising postharvest losses. However, several challenges remain. High equipment costs, process scalability, and optimisation for different food matrices need further attention. Additionally, ensuring uniform drying, integrating renewable energy sources into drying systems and maintaining food safety standards are critical areas for improvements. Future research should focus on optimising process parameters and developing hybrid technologies that combine the strengths of multiple approaches. Furthermore, advance real-time monitoring tools for better control of drying kinetics is needed. The use of Artificial Intelligence will be somewhat central to these novel adoptions. Overall, novel drying technologies embrace strong potential to transform the food industry by enabling high-quality, energy-efficient, and consumer-driven products, but more work need to address cost-effectiveness, scalability, and integration into existing processing systems.

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Spray Drying: A Promising Technique in Food Processing – Concept, Applications, and Latest Advancements

Nidhi Attri, Sukhpreet Kaur and Poonam Aggarwal

Abstract

The chapter explores the utilization of spray drying technology in food processing, focusing on its effectiveness in preserving nutrient quality, extending shelf life, and enhancing the functionality of food ingredients. Spray drying is widely used technique in the food industry due to its ability to convert liquid feed into stable, dry powder with minimal bioactive and mineral loss making it ideal for sensitive nutrients such as vitamins, antioxidants, and essential oils. This chapter presents principle of spray drying, covering process design, equipment, and operational parameters that impact product quality and advancements in a spray drying system. Advance spray drying and encapsulation methods offers precise control over particle size and distribution, significantly improving the delivery of nutrients in functional foods and nutraceuticals. Compared to conventional drying methods, nano spray drying in particular addresses the challenge of encapsulating small particles and preserving ingredient bioactivity. Despite limitations in scaling these advance technologies to industrial levels, ongoing innovations show promise in overcoming these hurdles. The chapter concludes with an overview of the commercial viability of spray drying and its role as an essential technology in the development of stable, nutrient-rich food products.

Keywords: drying, encapsulation, powder, bioactives, flowability

1. Introduction

Spray drying is an advanced dehydration technique widely used in the food industry due to its ability to produce free-flowing, stable with preserved nutritional quantity powders. This technique involves atomizing a liquid feed into droplets and exposing then to a stream of hot air, resulting in rapid moisture evaporation and powder formation [1]. The process is valuable for food processing industries such as dairy, fruit, and vegetable powders, encapsulated bioactives and instant beverages. Furthermore, this method is ideal for heat-sensitive materials, as atomized droplets

have a limited exposure time to hot drying medium due to their high surface to volume ratio. Additionally, evaporation after drying provides cooling effect [2–5]. Spray drying has evolved significantly as a result of constant innovation, including unique techniques to increase energy economy, product stability, and powder properties.

This technique was first mentioned in a U.S. patent published in 1872, where Samuel Percy described it as “an exposure of atoms to currents of air or other gases” while investigating the drying of dextrin, starch, and gelatin solutions to produce powders with reduced chemical changes and moisture content [6]. Although its industrial application began in the 1920s, the post-World War II dairy’s surplus milk production significantly advanced spray drying technology [7]. Over the years, technical advancements have resulted in improved control over particle shape, moisture content, and encapsulation efficiency, making spray drying the ideal method for generating high-quality food powders with low loss of bioactive components.

Recent advances in spray drying technology have centered on increasing efficiency and sustainability. Nano spray drying and other innovations enable the creation of nano-encapsulated bioactives with better solubility and tailored distribution [8, 9]. Furthermore, electrostatic spray drying and spray freeze drying have been developed to improve particle size distribution and powder reconstitution qualities. The combination of computational modeling and artificial intelligence (AI) has helped to improve process efficiency by anticipating drying kinetics and improving product uniformity [10].

Despite several benefits, this technique has some limitations, and spray dryer designs often utilized are inadequate to address these challenges. For instance, drying particularly products with high sugar content requires intermittent sweeping and hammering to remove sticky or deposited particles from wall surfaces of dryer [11]. Low-molecular-weight compounds such as invert sugars have high hygroscopicity, thermoplasticity, and low glass transition temperature (T_g), which contributes to this stickiness [9, 12, 13]. Other problems are degradation of interface-sensitive compounds like proteins [8], low powder recovery [14], high energy demand, and low energy efficiency [7]. To address these restrictions, researchers worldwide are exploring process improvements at various stages. The article presents the findings of research that adjusted the process to meet changing course and product needs, as well as the classic spray drying method’s features. The technology’s uses in the food business, such as microencapsulation of health-promoting compounds, need specific considerations for the process and product qualities.

Thus, the purpose of this chapter is to present: the foundation and principles of the spray drying process; industrially realized solutions; and system components. Recent reviews of spray drying have focused on specific product, such as fruit and vegetable juices [9], probiotics and food-grade bacteria [15], encapsulated oils [16], high-protein dairy powders [17], or stages of the process, such as atomization technologies in dairy.

2. Principle and components

Spray drying is based on the rapid evaporation of solvent from atomized liquid droplets in a controlled atmosphere, which produces dry powder particles [8]. The process is based on mass and heat transfer, with a liquid feed transformed into tiny droplets and treated to a hot drying medium, often air or nitrogen. Rapid moisture removal guarantees that the substance being dried retains its important

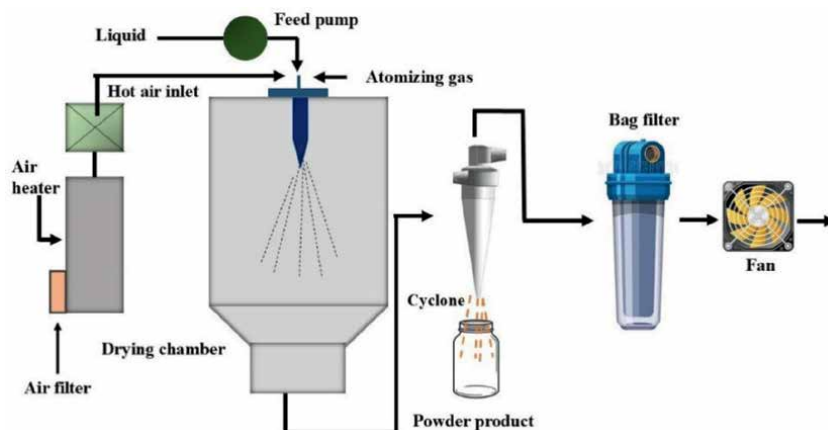


Figure 1.
 Components of spray drying system.

physicochemical and functional qualities [13]. Components of spray drying system have been presented in **Figure 1**.

The key steps involved in spray drying process include:

2.1 Feed preparation

The liquid feed, which can include a solution, suspension, or emulsion, is designed to meet the specified composition, viscosity, and stability. This might include dissolving or dispersing active substances, changing pH, adding stabilizers or encapsulating agents, and homogenizing to guarantee homogeneity and prevent phase separation [16]. The feed is then filtered to eliminate contaminants before entering the drying machine. Spray drying is 4–5 times more cost-effective than freeze drying [18], but has low thermal efficiency due to hot air circulating in a wide chamber without interaction with spray droplets [8, 19]. According to Barańska et al. [20] and Cheng et al., [8], the heat efficiency of spray drying ranges from 25 to 60%, with some even achieving <10% on a trial size. As previously stated, spray drying is also widely employed for encapsulation, in which a core substance is covered with a protective matrix (wall material) to allow controlled release when needed. Encapsulation protects bioactive substances from environmental variables such as high temperatures, humidity, oxygen, pH changes, and UV radiation [16, 21]. Several authors have reported encapsulating processes, such as extrusion, coacervation, and drying, and have been investigated for the preservation of sensitive chemicals [14, 22, 23]. However, because of its brief contact time with the hot drying medium, spray drying is perfect for encapsulating thermolabile compounds such as nutraceuticals, probiotics, flavors, and enzymes [24]. The encapsulation procedure necessitates precise emulsion preparation, particularly when working with non-water-soluble core materials [25]. The core is dispersed within the wall material, which is then homogenized with or without emulsifiers, depending on the qualities of the coating and encapsulated substance. Factors influencing the final capsule size include emulsion preparation process, wall material type, viscosity, and spray drying operating parameters [7]. Therefore, modifications in feed material preparation impact both energy usage during drying and powder qualities.

2.2 Atomization

An atomizer is used to break down the prepared feed into tiny droplets typically in diameters ranging from 2 to 600 μm , which considerably increases surface area and promotes effective moisture removal [18]. Pressure nozzles, rotary disk atomizers, and ultrasonic atomization are all methods of atomization that influence droplet size distribution and drying kinetics, respectively [7]. Different types of atomizers have been discussed in **Table 1**. Among them basic atomizers used in food industry are wheel atomizers and pressure nozzles [1]. In handling bulk quantities (40 t/h), rotatory atomizers are used, and control is relatively easy by speed control and eliminates the need for high pressure feed pump [26]. The major disadvantage of this type of atomizer is wall deposits typically originating from wall spray and high energy consumption and maintenance [27]. However, in pressure nozzles there is less energy consumption along with lower wall deposits. The liquid transmits from a nozzle orifice of the dimension 0.2 m with pressure ranging up to 1 to 30 mPa [28]. On the contrary, the capacity of this type of nozzle is limited and certain persisting disadvantages such as blockage and clogging. Moreover, pressure nozzles cannot be used for high viscosity liquids or containing suspended particles [27].

Another type of nozzle suitable for high viscous, abrasive liquids are pneumatic nozzles which creates finer particle distribution. However, this type of nozzle system has high energy consumption [28].

Conventional atomization processes cannot generate submicron-sized particles, which are critical for increasing the bioavailability of encapsulated compounds [27]. Furthermore, homogeneous heating and moisture evaporation are crucial, especially for thermolabile chemicals, because excessive droplet polydispersity might cause

Atomizer type	Principle	Advantages	Limitations	Applications
Rotary atomizer	High-speed rotating disk throws liquid tangentially, creating a fine spray.	High throughput, handles viscous feeds, robust.	Larger droplet size distribution, potential for product degradation due to shear forces.	Milk powder, coffee, fruit juices, detergents.
Pressure nozzle atomizer	High-pressure liquid is forced through a small orifice, breaking into droplets.	Smaller droplet size, good for heat-sensitive materials.	Lower throughput, may require high pressure pumps.	Pharmaceuticals, flavors, enzymes.
Two-fluid nozzle atomizer	High-velocity gas stream atomizes the liquid.	Flexible droplet size control, suitable for heat-sensitive materials.	Can be more complex to operate and maintain.	Fragile materials, encapsulated products.
Ultrasonic atomizer	High-frequency sound waves create vibrations, breaking the liquid into fine droplets.	Very small droplet size, uniform particle size distribution.	Low throughput, expensive.	Nanoparticles, specialized applications.

Table 1.
Types of atomizers used in spray drying.

overheating of smaller particles or insufficient drying of larger ones [1]. To overcome this issue, sophisticated nozzles such as electrostatic and ultrasonication nozzles are used, which provide greater droplet size control and encapsulation efficiency. Ultrasonication nozzles produce finer droplets by high-frequency vibrations, and electrostatic nozzles use electrical forces to manufacture precise droplets, both of which considerably improve spray drying effectiveness [29, 30].

2.3 Drying and evaporation

After atomization, the tiny droplets are introduced into a drying chamber where they come into contact with heated air, allowing for fast moisture evaporation. The drying kinetics rely on:

- a. Temperatures at the air intake and outflow (e.g., 150–200°C for the inlet and 60–80°C for the output in dairy powders).
- b. Airflow pattern (co-current versus counter-current drying).
- c. Humidity and feed composition effect droplet shrinkage and powder shape.

Conventional spray dryers have intake air temperatures ranging from 150 to 220°C, whereas dried products in food applications might reach 70–90°C. However, such high temperatures can destroy sensitive components such as lycopene, β -carotene, anthocyanins, and vitamin C [23]. In addition to that, numerous authors have reported that controlling drying temperature reduces nutrition loss while assuring effective moisture removal [31–33]. For instance, high temperatures can damage heat-sensitive bioactives, whereas low temperatures can cause significant residual moisture and poor shelf stability. Another limitation is that drying fruit and vegetable juices, molasses, and honey need a carrier agent due to their low glass transition and stickiness [18, 20] which causes wall deposition issues and drying challenges. Researchers have developed innovations to reduce drying temperatures for certain uses, including vacuum and dehumidified air.

2.4 Particle formation and separation

As the moisture evaporates from the atomized droplets in the drying chamber, solid particles form. Several factors impact the properties of these particles, including the proportion of solids in the feed, the surface makeup of the droplets, and the particle's ability to agglomerate. A higher solid concentration in the feed solution results in denser and more uniform particles, whereas lower concentrations may produce irregularly shaped or fragmented powders [19]. Goula and Adamopoulos, [34] reported that an increase in feed solid concentration from 14 to 22% in tomato powders led to a higher insoluble solid content, resulting in larger particle size and more irregularly shaped particles. Moreover, the surface composition of the drying droplets also influences the wettability and reconstitution qualities of the finished product [19]. For example, a powder with a hydrophobic surface may have low solubility, which is undesirable in many food applications. Furthermore, agglomeration or the tendency of particles to cluster together impacts the powder's flowability and dispersibility [35]. Excessive use might cause clumping, making the substance difficult to handle or dissolve.

To address these difficulties, researchers have concentrated on optimizing particle creation using wall materials and method improvements. Arumugham et al. [36] found that using gum Arabic and maltodextrin as an encapsulating ingredient improves date powder flow characteristics and reduces stickiness, which is a typical issue when spray drying high-sugar or bioactive-rich formulations. Researchers want to increase stability of spray-dried powders along with solubility and overall quality by carefully choosing wall materials and adjusting drying conditions.

Once the dried particles exit the drying chamber, they are carried by the airflow into a separation system, typically consisting of cyclone separators, bag filters, or electrostatic precipitators. The recovery technique is based on particle size distribution, with bigger particles settling quicker and tiny ones staying airborne longer. Cyclone separators use centrifugal force to efficiently separate fine powder from the drying air, while bag filters trap any ultrafine particles that escape [37]. Electrostatic precipitators employ electrically charged plates to attract and collect tiny powder particles, reducing waste and increasing collection efficiency [38]. The separation technique used is determined by the parameters of the powder, such as particle size, density, and flow behavior. Moreover, cyclones have lower efficiency for particles smaller than 15 μm , whereas bag filters are ineffective for particles smaller than 2 μm [7]. Conventional separators cannot separate tiny particles or split them depending on size, resulting in a powder with a variety of sizes.

2.5 Direction of material flow in spray drying system

Material flow in a spray drying system might take the form of concurrent, counter-current or mixed flow processes, each having their own unique properties. In the concurrent flow process, both the crushed liquid material and the drying air flow in the same direction, arriving from the top and departing from the bottom of the drier [39]. According to Quintero et al. [40], this configuration exposes the droplets to higher drying air temperatures, which causes fast evaporation and produces particles with low density and hollow structures. In the counter-current flow method, the liquid material and drying air travel in opposing directions through the chamber. This approach subjects virtually dry particles to higher temperatures, frequently producing extremely dry products [41]. The mixed flow method incorporates characteristics of both, with the material atomized upward and the drying air descending from above. The material first flows upward but is finally carried downward by the airflow, both of which escape out the lowest half of the chamber. This method is favored when chamber space is restricted [42].

3. Applications in food processing

Spray drying has several commercial uses, including powder formulations and aerosols in the chemical, food, and pharmaceutical industries. Industrial spray drying systems may evaporate up to 40 tons of water per hour, depending on process and production requirements [7]. Global leaders in spray drying equipment include GEA Group (Germany), SPXFLOW - ANHYDRO (USA), DEDERT Corporation (USA), Tetra Pack (Sweden), and Lemar Drying (China) [7].

This process offers numerous benefits, including producing free-flowing, high-quality materials with excellent particle size, flow, density, moisture content,

solubility, and instantanization properties. It is also suitable for both heat-resistant and heat-sensitive materials. Spray drying is ideal for food industry applications since it protects tastes, colors, nutritional value, and other functional aspects. This is vital in applications including baby meals, coffee powders, taste and color additives, and pharmaceutical chemicals. Applications of spray drying in food processing have been discussed in **Table 2**.

Core material	Wall material	Process parameters	Results	References
Production of dried powder				
Cagaita (<i>Eugenia dysenterica</i> DC.) fruit extracts	Inulin or gum Arabic (10, 20, and 30%).	Inlet temperature (120, 140, and 160°C), a peristaltic pump at a fixed rate of 0.60m ³ /min and nozzle diameter 1.2 mm.	Increase in yield, solubility, and glass transition temperature. Decrease in water activity and hygroscopicity with increase in concentration of wall material. With increase in inlet temperature, an incline in yield and hygroscopicity was observed.	[43]
Sweetened yogurt powder	Maltodextrin	Inlet air temperature (140–180°C), feed flow rate (0.3–0.6 L/h), nozzle diameter: 0.5 mm	Results indicated that the corresponding response surface model was sufficient to describe and predict powder characteristics in terms of moisture content (% db), bacteria survival ratio (N/No), acetaldehyde retention (%), and overall sensory score of 98.24, 98.44, 98.57, and 97.85%, respectively. Survival ratio: 2.37×10^3	[44]
Pomegranate juice	Maltodextrin (25, 35 and 45% w/w)	Inlet air temperatures (124 and 143°C), outlet temperature: 48–70°C, feed rate: 0.5–1.5 kg/g ⁻¹	Nutritionally rich pomegranate juice powders were produced at 25% maltodextrin level and 143°C, with a low moisture content of 3.19%, high ash content of 0.58%, safe pH value of 3.4, and considerable total phenolic compounds of around 903 mg gallic acid/g db.	[45]
Mango pulp	Maltodextrin	Inlet temperature of 150°C, flow rate of 0.42 L h ⁻¹	Improved surface morphology, stickiness, and solubility	[46]
Encapsulation of flavors and functional food ingredients				
Ghee flavor	Guar gum, whey protein concentrate	Inlet temperature: 160°C, outlet temperature: 45–50°C, feed flow rate: 5 ml/min, nozzle diameter: 1.5 mm	Yield of 53.2%.	[47]
Strawberry flavor	Maltodextrin, gum arabic, modified starch, soluble fiber, β -cyclodextrin	Inlet temperature 130–190°C, Outlet temperature: 80°C, feed flow rate: 5–8 ml/min,	Process yield ranged from 44.04 to 86.61%.	[48]

Core material	Wall material	Process parameters	Results	References
Vanilla extract	Modified rice starch	Inlet air temperature (100–140°C), outlet temperature (70–90°C)	Achieved 86.2% encapsulation	[49]
<i>Lactobacillus plantarum</i>	Agave fructans, aloe vera mucilage	Inlet air temperature, 150°C, outlet temperature was 78–90°C, flow rate, 0.8 L/hr.;	Survival rate of 70%	[50]
<i>Lactobacillus acidophilus</i>	Pectin, milk powder, maltodextrin	Inlet temperature: 170°C, feed flow rate: 30–50 ml/min, nozzle diameter: 0.5 mm	Survival rate ranged from 45.26 to 81.40%	[51]
Oleoresin encapsulation				
Ginger oleoresin	Skim milk powder and maltodextrin	Inlet temperature: 170°C, outlet temperature: 70°C, feed flow rate: 15 ml/min.	Maximum 92.31% of encapsulation efficiency of oleoresin was achieved.	[52]

Table 2.
Application of spray drying in food processing.

3.1 Drying

Spray drying is a common technology in the food processing for transforming liquid or semi-liquid foods into stable, free-flowing powders. This process includes atomizing a liquid feed into thin droplets, which are then quickly dried with hot air to remove moisture while retaining the product's nutritional, functional, and sensory qualities. Spray drying is often used to manufacture dairy powders (such as milk, whey, and casein), fruit and vegetable powders, coffee and tea powders, and flavor capsules.

High perishability is a major challenge in fruit and vegetable production [53, 54]. Postharvest spoilage leads to rapid quality deterioration, resulting in significant waste. The production of fruit and vegetable juice powders is a promising strategy to minimize loss of perishability, packaging, storage compared to its liquid counterpart, allowing fruits and vegetable juices to be preserved for weeks to months [55].

One of the main advantages of spray drying in food applications is its ability to extend shelf life by decreasing moisture content, thereby minimizing microbial growth and enzymatic degradation [56, 57]. Additionally, it enhances ease of handling, storage, and transportation compared to liquid products. However, spray drying efficiency and product quality depend on several factors, including feed composition, drying temperature, atomization parameters, and carrier agents [58–60]. High temperatures can degrade sensitive bioactive compounds, such as vitamins and polyphenols, necessitating optimized processing conditions [56, 61]. The use of carrier agents like maltodextrin, gum arabic, or starch is common in spray drying fruit juices and plant extracts to improve powder stability, prevent stickiness, and enhance solubility.

The applications of spray drying have been discussed below:

3.1.1 Milk and dairy powders

Spray drying is often used to produce milk powders, such as whole milk, skim milk, and whey powders. It provides prolonged shelf life, ease of transportation, and reconstitution qualities [62]. Camci [63] reported that by optimizing spray drying parameters such as inlet temperature, pressure, and humidity, an overall efficiency of 75.01% can be obtained in milk powder. This technique is highly effective for drying dairy products, offering significantly shorter processing times compared to alternatives such as freeze drying or vacuum-drying [64]. One of the key advantages of spray drying is its ability to provide direct, one-step control over particle production and size distribution [64].

The quality and properties of spray drier dairy products such as water content, insolubility index, caking tendency, and moisture content are influenced by critical processing parameters, including temperature, air flow, and liquid flow rate [65]. In another study, Philipp et al. [66] presented an innovative spray drying design for drying milk using a plant-wide heat integration method with a precise and optimal model.

Several factors influence spray drying performance of dairy liquids, including total solids content, viscosity, protein and fat composition, and preheat treatment conditions [64, 67]. For instance, Bista et al. [60] reported that as total solids concentration increases, the viscosity of skim milk concentrate increases nonlinearly. This phenomenon is likely due to intensified micelle-micelle interactions, and an increased volume fraction of dispersed particles as water is removed.

Another challenge in spray drying of dairy concentrates is age thickening, which refers to gradual increase in viscosity during storage. The main factors which contribute to age thickening include total solid content, storage temperature/time combination, shear rate, and salt balance changes [60]. Age thickening increases with increased total solid content and temperature [60].

3.1.2 Fruit and vegetable powder

Spray drying liquid fruit and vegetable purees yield powders with flavor, color, and nutritional value. These powders are included into drinks, desserts, and nutritional supplements [68–70]. Barbosa et al. [71] studied drying methods for probiotic orange powders and reported that the spray drying (SD) and freeze drying (FD) retained higher vitamin C levels, with FD offering better color retention without maltodextrin. However, due to FD's high costs and long drying times, SD emerged as a cost-effective option for producing functional probiotic orange juice powders. Several studies have examined the impact of different carrier materials on product yield. Research indicates that maltodextrin (77%) is more effective carrier agents than gum Arabic (68%) for drying of sour cherry [59]. Additionally, higher concentrations of carrier agents have been consistently linked to improve product yield. This positive correlation has been observed in various fruit juices, including tamarind juice [72], raisin juice [73], orange juice [74], pomegranate juice [75], black mulberry juice [58], sugarcane juice [57], and sucrose solutions [76].

3.1.3 Coffee and tea powders

Spray drying is used to manufacture instant coffee and tea powders, preserving taste and fragrance while allowing for fast rehydration. Ghirişan and Miclăuş [77] compared physical and antioxidant properties of soluble coffee dried using two

drying techniques, i.e., spray and freeze drying. Authors reported that spray-dried coffee exhibited a more uniform particle distribution, whereas freeze-dried coffee retained a higher concentration of antioxidants. In another study, Padma Ishwarya et al. [78] explored an innovative approach, spray freeze drying for coffee powder production. The authors reported the highest retention of volatiles, i.e., 93% of those present in feed. The resulting coffee powder exhibited instant solubility and a porous structure.

Several parameters significantly influence the efficiency and quality of spray drying tea and coffee granules, including feed concentration, inlet and outlet air temperatures, atomization settings, and carrier agent. Feed concentration influences the viscosity and flow characteristics of the liquid extract, with more solids content enhancing yield but potentially increasing clogging [61]. Temperature settings are critical, as high inlet temperatures can cause thermal degradation of polyphenols and volatile aroma compounds in tea [79], whereas low drying temperatures can result in poor powder stability and increased moisture retention in coffee [61]. Thi Anh Dao et al. [56] reported that the optimal spray drying parameters for best retention polyphenols were of 135.8°C, 6.8 rpm, and 10.3% dry basis of input flow resulted in a TPC of 312.96 mg GAE/g dry basis of tea powder product. Atomization conditions, such as nozzle type and spray pressure, affect particle size distribution, with finer droplets resulting in superior reconstitution qualities in instant tea and coffee [61].

The wall material used in the spray drying process plays a significant role on the qualities of instant tea powder. It influences the microstructure of the produced microcapsules [27]. Numerous studies have shown that the size, shape, and surface features of microcapsules are determined by the qualities of the wall material [61, 80]. Various authors have reported production of instant tea powders through microencapsulation combined with spray drying [56, 81, 82]. Eroğlu et al. [81] formulated tea powder with a blend of rosehip fruit and hibiscus using spray drying technique with an inlet temperature of 130°C, outlet temperature of 85°C, and 18.4% maltodextrin as a carrier agent.

3.1.4 Protein powders

Spray drying is one of the most effective and extensively used ways for producing protein powders because it can rapidly turn liquid protein solutions into stable, free-flowing powders with minimum functionality loss. This process includes atomizing a liquid protein feed into small droplets, which are subsequently exposed to hot air, causing fast water evaporation and producing dry powder particles. Spray drying is a standard method for producing whey protein, soy protein, pea protein, and casein powders, which are widely utilized in the food, pharmaceutical, and sports nutrition sectors [83, 84].

There are various factors which influence clumping in these protein powders which include bulk composition of particles (such as fats and proteins), physical properties (size and shape, surface roughness, and density), and environmental and processing conditions [85–87]. Both et al. [88] investigated particle distribution and properties of spray dried maltodextrin and whey protein mixtures. The authors reported that optimizing the inlet temperature and humidity in the spray dryer increased the number of hollow particles from 32 to 59% in a mixture containing 75% whey protein powder and 25% maltodextrin, while reducing the wrinkled particles to 34 to 19%.

Various substances that can be used in spray drying to stabilize protein powders include amino acids, glycerols, polymers, and saccharides [89]. Amino acids play

a crucial role in stabilizing both liquid and solid protein formulations. It has been proposed that amino acids enhance the stability of protein powders by filling “free volume holes” in amorphous formulations [90]. Additionally, charged hydrophilic amino acids contribute to stabilization through hydrogen bonding and antioxidant properties [89]. The use of amino acid buffers has also been explored in spray drying monoclonal antibody proteins, with a recent study indicating 6% histidine buffer is more effective than lactate in preserving integrity of an Immunoglobulin monoclonal antibody [89, 91].

Glycerol is an important substance, and the method by which it stabilizes big macromolecules such as proteins is poorly understood. Although glycerol is a plasticizer but it has been observed that it restricts secondary or local molecular movements, thereby contributing to stabilization [92].

Throughout the spray drying process, protein powders are subjected to a variety of thermal, interfacial, and mechanical stresses, transitioning from a bulk liquids state to dried particles [17]. During drying, the temperature of the atomized feedstock quickly rises, until the surface of the droplets maintains 100% relative humidity in the system [89]. In the literature reviewed analyzed average inlet temperatures ranged from 45 to 225°C.

3.1.5 Spices and flavor powders

Spray drying is widely used to encapsulate essential oils and flavors, resulting in stable powders that are resistant to oxidation and volatilization. Oleoresins are extracted from spices like turmeric, ginger, fennel, and paprika and encapsulated with zein and skim milk powder (**Table 2**). Oleoresin derived from fruits, such as tomatoes, was encapsulated using two processes, i.e., spray and freeze drying. Spray-dried oleoresins had an encapsulation effectiveness of 71 to 99.9%, whereas lyophilized oleoresins had an efficiency range of 17–74% [52, 93, 94]. This clearly shows that spray drying effectively encapsulates oleoresins. Encapsulation of some of the flavors has been summarized in **Table 2**.

3.1.6 Powdered starch and carbohydrate products

Spray drying is used to turn starch and carbohydrate liquids into powders for use as food thickeners and sweeteners. Carbohydrates play crucial role as wall material in spray drying process, influencing both powder characteristics and encapsulation efficiency. Ding et al. [95] investigated impact of seven different carbohydrate, including two non-reducing disaccharide (sucrose and trehalose), one fructose polymers (inulin), three glucose polymers (maltodextrins with different DE), and modified starch on the powder characteristics and encapsulation efficiency of micro-encapsulated lutein. The authors reported higher product yield using modified starch and maltodextrins, while superior encapsulation efficiency was achieved using inulin, trehalose, and modified starch as a wall material.

3.2 Encapsulation of bioactive compounds

Spray drying is an effective encapsulation method for safeguarding delicate bioactive substances like vitamins, antioxidants, and essential oils from environmental influences such as light, heat, and oxygen. This encapsulation protects the functional qualities of these drugs while also increasing their stability and bioavailability.

Nambiar et al. [96] reported 94.86% encapsulation efficiency for total phenols in tender coconut water using spray drying technique (inlet temperature 120°C), with *Moringa Oleifera* (1.5%) and maltodextrin (30%) as coating material. Research suggests that spray-dried oils are more effective in encapsulating bioactive lipids (about 60–97%) than freeze-dried oils (56–90%) [7].

One major factor that affects encapsulation efficiency in spray drying system is the inlet temperature [97]. Lower intake temperatures reduce evaporation rates, resulting in increased moisture content in microcapsules [98]. Boyano-Orozco [99] found that encapsulating bioactive chemicals from rambutan peel at 160°C resulted in a moisture content of $3.95 \pm 0.10\%$ and an 85% encapsulation efficiency. Another factor which influences encapsulation efficiency is cycle mode. The cycle mode of a spray drying system varies depending on the type of solvent being used. For organic solvents, a closed-loop setup with a condenser is recommended to reduce the risk of explosion at high temperatures, reduce the vulnerability of the final powder to oxidation, and prevent the release of organic solvents into the environment. The condenser collects the organic solvents. To dry water-based feedstock, an open-loop configuration is used. In this case, using a condenser is not necessary [100].

3.3 Production of functional food ingredients

Functional foods, such as bioactive components, probiotics, prebiotics, and dietary fibers, aim to prevent chronic diseases and improve overall health, in addition to providing basic nutrition. The capacity of spray dryer to turn liquid extracts into powdered forms has proven useful in the production of beneficial food components.

One of the most effective approaches for developing functional foods is the encapsulation of nutraceuticals and phytochemicals in delivery systems, ensuring their stability, safety, and controlled release at the desired place. For example, oil encapsulation via spray drying has been widely explored to improve handling qualities and oxidative stability [101–103]. Bhushan et al. [104] reported 73.10–86.17% encapsulation efficiency using spray drying in soy protein isolate-jack fruit seed starch with flaxseed oil as an encapsulating material. The resulting encapsulated powder not only retained the nutritional benefits of jackfruit but also incorporated the antioxidant properties of flaxseed oil.

Probiotics as functional foods are vital for gut health as they eliminate pathogenic microorganisms. Spray drying is considered as one of the most effective ways for producing probiotics, as it allows for bulk manufacturing at a lower energy cost. Additionally, this technique also holds potential for encapsulating bacteria in diverse protective matrices to enhance their resistance to storage, technological procedures, and digestive stressors. Various studies have demonstrated successful spray drying encapsulation of bacteria such as *Lactobacillus plantarum*, *Lactobacillus Acidophilus*, and *Lactobacillus bulgaricus* for the production of functional probiotics [44, 50, 51]. The application of spray drying in functional food ingredients has been summarized in Table 2.

3.4 Retention of nutritional, sensory, and color properties

Maintaining nutritional and sensory properties of a food products intact during processing is crucial. Spray drying has been demonstrated to efficiently preserve these qualities, making it an ideal method for drying heat-sensitive materials. Its rapid drying process minimizes nutrient degradation while retaining the original flavor and color.

For instance, Balci-Torun and Ozdemir [48] and Ocampo-Salinas et al. [49] have successfully explored the stabilization of strawberry and vanilla flavors, respectively, using spray drying with wall material such as maltodextrin, gum Arabic, and modified starch, achieving yield ranging from 44.04 to 86.2%. Furthermore, spray drying enables control release of flavors in food applications. Certain factors which influence this release profile include carrier material's thickness and weight, particle or core material size and shape, release medium, and encapsulation technique are all elements to consider [97]. Beyond flavor, spray drying is effectively used for color encapsulation. Ab Rashid et al. [105] and Patel et al. [106] have explored various color encapsulation processes using wall materials such as maltodextrin, gelatin, and whey protein isolate.

4. Advancements in spray drying system

Spray drying systems have advanced significantly in recent years, encouraged by advances aimed at increasing efficiency, product quality, and process control. Emerging technologies like ultrasonication, artificial intelligence (AI), computerized systems, and sustainable techniques are changing the way spray drying is used in the food processing and other sectors. Different types of advancements have been discussed in **Table 3**.

4.1 Ultrasonication in spray drying

Ultrasound pretreatment, a nonthermal procedure for modifying food components, has received attention in food science and technology because of its potential for the development of innovative, gentle, yet focused processes to improve the quality and safety of processed food [94, 95].

Ultrasonication technology is increasingly being used in spray drying systems to boost atomization efficiency and create consistent droplet sizes. This approach generates ultrasonic vibrations, allowing for the creation of finer, more homogenous particles, which are very useful for encapsulating bioactives and thermolabile chemicals. Jin et al. [107] found that ultrasonication improves powder solubility, dispersibility, and bioavailability by producing nanoparticles with a larger surface area. Furthermore, ultrasonication reduces nozzle blockage, resulting in more consistent performance and lower maintenance expenses. In another study, Jin et al. [107] investigated ultrasonic assisted spray drying for development of instant egg white powder. The authors reported that high intensity (20 KHz) ultrasonic treatment enhanced the wettability, dispersibility, and solubility of egg white powder by modifying its protein microstructure, reducing particle size and enhancing depolymerization of protein aggregates. These studies suggest that ultrasonic pretreatment can improve the instant properties of protein powders.

Another application of ultrasound in spray drying system is the use of ultrasonic nozzles. Ultrasonic nozzle atomizers generate droplets by high-energy vibrations. According to Selvamuthukumaran et al. [108], these approaches are often preferred over standard atomization methods for various purposes. The devices include a piezoelectric transducer with a spray tip, an ultrasonic generator, an external liquid applicator, a precise liquid distribution system, and air directors [108]. The transducer, powered by an ultrasonic generator and resonant at a certain frequency (35, 45, or 60 kHz), produces high-frequency sound waves or vibrations. When vibration

Technology	Principle	Advantages	Disadvantages
Ultrasonication	Utilizes ultrasonic waves to break down liquid into fine droplets, improving atomization and particle uniformity.	• Produces uniform, fine particles. • Enhances solubility, dispersibility, and bioavailability. • Reduces nozzle clogging.	• Requires additional equipment thus increases costs. • Limited scalability for industrial applications.
Artificial Intelligence and Machine Learning	Uses algorithms to analyze data, predict outcomes, and optimize spray drying parameters in real time.	• Precise control of drying conditions. • Reduces product variability and energy consumption. • Streamlines scale-up processes.	• High implementation costs. • Requires skilled personnel for operation and maintenance.
Computerized Process Control Systems	Integrates sensors and software for automated monitoring and control of spray drying parameters.	• Consistent product quality. • Real-time adjustments to prevent overheating or under-drying. • Enhanced traceability and compliance with standards.	• Dependence on high-quality sensors and software. • Risk of system failure, impacting production.
Nano Spray Drying Technologies	Employs advanced nozzles and atomizers to produce ultra-fine particles (below 1 μm) for improved functional properties.	• Ideal for encapsulating bioactives and pharmaceuticals. • Enhances bioavailability and controlled release. • Produces stable powders.	• Limited throughput compared to conventional spray drying. • High equipment and operational costs.
Electrostatic Spray Drying	Applies an electric field during atomization to improve droplet dispersion and particle formation.	• Better particle size control. • Higher encapsulation efficiency. • Suitable for sensitive bioactives like flavors and aromas.	• Requires specialized equipment. • Potential safety concerns due to high voltage operation.

Table 3.
Advancements in spray drying system.

energy is delivered to the resonant atomizing surface, it spreads and causes a thin liquid layer to develop at the tip of the atomizer.

4.2 Artificial intelligence and machine learning

Artificial intelligence (AI) and machine learning (ML) are transforming spray drying by allowing for real-time monitoring, predictive modeling, and process optimization. These technologies aid in accurately managing factors like as inlet and outlet temperatures, air velocity, and feed rate to create the desired powder properties [109, 110]. Przybył and Koszela, [111] discussed the application of AI-driven systems to anticipate moisture content and powder flowability, hence lowering product variability and energy usage. Furthermore, ML algorithms can assess past data to recommend appropriate drying settings, which simplifies the scaling-up process for industrial applications [112].

4.3 Computerized process control systems

Modern spray drying systems now have powerful digital controls that allow for automatic monitoring and modification of important parameters. These systems employ sensors and data analytics to assure consistent product quality while minimizing human interaction [113]. For example, adaptive control systems may dynamically alter drying air temperatures based on feed qualities, avoiding delicate substances from being over-dried or thermally degraded. Computerized systems also improve traceability and regulatory compliance, making them vital in the food and pharmaceutical industries [7]. Moreover, computer-controlled pumping and mixing system can automatically generate inlet feeds with programmable composition and reduce cross contamination to less than 3% [113].

4.4 Nano spray drying technology

Nano spray drying is a cutting-edge technology for creating ultra-fine powders with particle sizes under 1 μm . This method is very useful for encapsulating nutraceuticals, flavors, and pharmaceutical active ingredients that require high bioavailability and controlled release [114–116]. Vibrating mesh spray method effectively produces submicron particles for encapsulating vitamin B12, vitamin E, and folic acid, despite the longer process time [117]. Another study utilizing gum arabic and lecithin as natural emulsifiers produced eugenol nanocapsules with high re-dispersibility in water and retained their physicochemical characteristics after rehydration [118].

4.5 Energy efficient and sustainable designs

Sustainability has been a critical consideration in the development of spray drying systems. The carbon footprint of spray drying technique has been greatly reduced due to innovations in heat recovery systems, solar-assisted drying and energy efficient atomizers. Furthermore, ecological drying aids like as plant-based carriers and biodegradable wall materials are being researched to replace synthetic equivalents, boosting eco-friendly practices in powder production [119, 120].

4.6 Multistage and hybrid systems

Multi-stage spray drying systems, which include additional drying or chilling stages, increase powder flowability and moisture content management. Hybrid methods that combine spray drying with freeze drying, vacuum drying, or microwave drying are increasingly gaining popularity for preserving the functional and sensory qualities of heat-sensitive foods [121, 122]. Ishwarya et al., [123] found that spray freeze drying improved antioxidant retention in fruit powders compared to standard procedures.

4.7 Electrostatic spray drying

Electrostatic spray drying, which uses an electric field during atomization, provides better particle size control and encapsulation efficiency. This approach reduces bioactive loss during drying and enables the creation of powders with distinct surface morphologies appropriate for certain applications [124, 125]. Beaupeux et al. [126] emphasized its potential for capturing tastes and flavors, assuring sustained release in

food matrices. In another study, Jayaprakash et al. [124] compared electrostatic spray drying, spray drying, and freeze drying for dehydration of *Lactocaseibacillus rhamnosus* GG and reported that electrostatic spray drying preserved bacterial cells during drying as compared to spray drying and imparts better stability of cells during storage than freeze and spray drying.

These innovations increase the adaptability and effectiveness of spray drying, satisfying the needs for high-quality, useful, and sustainable powders in current food processing. As research and technology advance, spray drying systems will become more complex and adaptable to new industrial applications.

5. Conclusion

Spray drying has evolved as a flexible and effective food processing technology that converts liquid or semi-liquid feed into high-quality powders with longer shelf life and functional qualities. The method's versatility to different food matrices, as well as its ability to maintain sensitive bioactives under regulated drying conditions, have made it essential for the manufacture of dairy powders, fruit and vegetable concentrates, instant drinks, and nutraceuticals. Recent advances in spray drying systems, including as ultrasonication, nano spray drying, electrostatic atomization, and the incorporation of artificial intelligence and machine learning, have considerably improved accuracy, energy efficiency, and scalability.

Despite these benefits, disadvantages such as high energy expenditures, probable thermal damage of heat-sensitive materials, and equipment complexity remain. To address these restrictions, further innovation in sustainable methods, alternate drying aids, and smart process monitoring is required. By overcoming these problems, spray drying will continue to be a cornerstone of contemporary food processing, promoting the creation of innovative food items while assuring customer quality and safety. With continued research and integrative methods, this methodology will adapt to suit the food industry's ever-increasing demands while also enhancing global food technology.

Conflict of interest

None.

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Nanoencapsulation and Powder Delivery of Bioactive Compounds Using Nanotechnology and Freeze-Drying Techniques

Dilara Konuk Takma

Abstract

One of the increasing applications of nanotechnology in food science is the nanoencapsulation of bioactive compounds. Nanoencapsulation is desired to increase the solubility, bioactivity, and bioavailability of bioactive compounds or to protect them against environmental conditions. However, bioactive compounds encapsulated in various nanosuspensions need to be converted into further forms to maintain their stability for long-term storage. Therefore, it is important to stabilize the product while preserving its functional properties through drying technologies. Freeze-drying technique is an important final step in nanoencapsulation because it stabilizes nanoscale materials by maintaining their structure and properties. Freeze-drying technology is used to remove solvents from nanoparticle suspensions developed for the transport of bioactive compounds. Thanks to this technology, stable nanopowder products can be obtained to deliver bioactive compounds. This chapter summarizes advances in the applications of the freeze-drying technique to obtain nanoencapsulated powders, encapsulation efficiencies, and bioactive properties of freeze-dried nanoencapsulated powders incorporating phenolic compounds, essential oils, and oils rich in polyunsaturated fatty acids. The chapter emphasizes the significance of nanoencapsulating bioactive compounds using nanotechnology and delivering them in powdered form using freeze drying, highlighting their potential and innovative applications in the food industry.

Keywords: nanoencapsulation, lyophilization, nanopowders, bioactive constituent, functional properties

1. Introduction

Bioactive compounds are mainly known as the components found in foods and consumed naturally or present in dietary supplements taken in the body to meet the required level to provide health functions. These compounds can protect the body against diseases and metabolic disorders thanks to their wide range of functional properties. Their most important known functions include antioxidants,

anti-inflammatory, and anticarcinogenic effects. Fruits and vegetables constitute the most important source of bioactive compounds. Of these, phenolics, flavonoids, anthocyanins, tannin, betalain, and carotenoids are produced as secondary metabolites in various fruits and vegetables [1]. Today, new diet approaches are being developed to prevent common chronic diseases. For this purpose, functional foods, which differ significantly from traditional foods, offer positive effects in terms of health by using bioactive compounds [2]. Nowadays, the design of new functional food products is increasing in order to deliver bioactive compounds to consumers while preserving their functional properties. In addition, efforts are being made to improve the properties of bioactive compounds such as solubility, transportation to the targeted area in the human body, and controlled release in order to treat some diseases. This is possible with advanced carrier systems in which bioactive compounds are encapsulated [3].

It is important to convert bioactive compounds into more stable forms due to their sensitivity to environmental factors such as temperature, oxygen, and light. Technological developments are expanding the opportunities for developing dietary supplements or functional foods by protecting these compounds from environmental factors and stabilizing them. Freeze drying is one of these technologies and is the most suitable method for converting bioactive compounds into powdered product form. Freeze-drying encapsulation technique is of interest because it can best preserve the properties of bioactive compounds compared to spray drying. Studies have shown that bioactive compounds are encapsulated with high encapsulation efficiencies (>85%) by freeze drying [4]. Nanoencapsulation is used for many potential applications by encapsulating bioactive compounds at the nanoscale. The primary goal is to protect active compounds such as polyphenols, enzymes, antioxidants, and vitamins from adverse environmental conditions. However, the use of nanotechnology for controlled release at targeted sites is also increasing. Freeze drying plays a role here to provide a high degree of stabilization and is used to obtain an acceptable product by maintaining the physicochemical stability of the nanoparticles during storage conditions [5].

This chapter aims to examine the potential of nanotechnology and freeze drying together in the nanoencapsulation of bioactive compounds and their presentation in powder form. The importance of nanosystems for the efficient delivery of bioactive compounds, the role of freeze drying in providing stability in nanosystems, encapsulation efficiencies and bioactive properties of freeze-dried nanoencapsulated powders are discussed.

2. Nano-system design for encapsulation of bioactive compounds

Bioactive compounds can be incorporated into foods with a wide range of functional properties such as antioxidant, anti-inflammatory, and antimicrobial. However, bioactive compounds face limitations such as low solubility and bioavailability in food systems. Nanoencapsulation technology can increase the solubility and stability of these compounds by reducing their sensitivity to external effects such as heat, light, and oxygen. In addition, it improves the absorption of compounds into cells by protecting them from external factors and thus expands their areas of use [6]. Moreover, bioactive compounds are highly active compounds and may lose their functional effects due to exposure to oxygen, light, high temperature, pH, and various environmental conditions. Therefore, encapsulation technology is also used

to preserve activities to obtain the highest functional effect [6]. Different encapsulation approaches are applied in food science to protect the bioactive compounds from external factors. In addition to protecting the bioactive compound in the encapsulation process, enhancing the properties of the compound and providing controlled release are possible with the nanoencapsulation technique. Nanoencapsulation generates particles at nanoscale, and these nanoparticles show high activity due to their increased surface/volume ratio. Thus, they offer innovative applications in various fields [7]. Nanotechnology is increasingly being applied in the food industry. The main ones are improving some sensory properties of foods such as color, texture, and taste; extending storage time with preservative effects; providing functional properties to food packaging systems; and developing functional foods with efficient absorption and solubility [8].

Main features of nanoencapsulated bioactive compounds and effective factors on these features are presented in **Figure 1**. The ability of bioactive components to provide functional effects in nanoencapsulated form depends on the properties of the obtained nanoparticles, which are primarily particle size, polydispersity index, zeta potential, encapsulation efficiency, and controlled release (**Figure 1**). These properties determine the chemical, physical, and functional behavior of nanoparticles and are important parameters that need to be optimized according to the applications. For example, size and shape of the particles affect the particle's ability to reach the target, while surface functionality and charge determine the surface interactions and stability of the particles. The techniques used for nanoencapsulation include emulsification, solvent evaporation, spray or freeze-drying, coacervation, inclusion complexation, nanoprecipitation, and supercritical fluid techniques. The selected technique, variables, and type of materials have an effect on the main properties of nanoencapsulated bioactive compounds [7, 9].

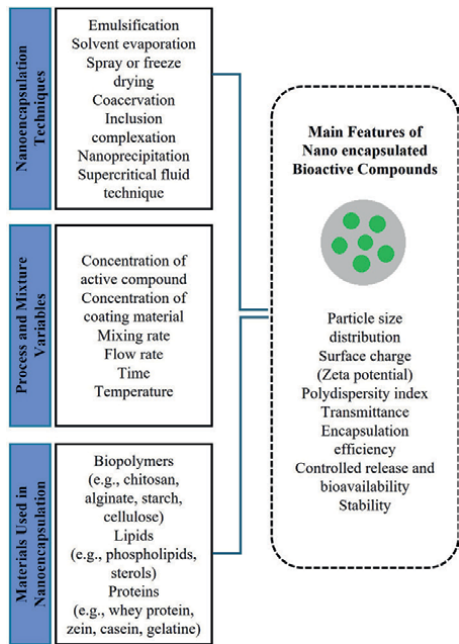


Figure 1.
Effective factors on features of nanoencapsulated bioactive compounds.

Nanoparticles can have different shapes and structures such as spherical, cylindrical, tubular, conical, hollow-core, and spiral. Their sizes can vary between 1 and 100 nm. Particle size distribution is one of the most fundamental and important measurements for nanoparticle characterization. Particle size and distribution in the solid phase are most widely measured using Scanning Electron Microscope and Transmission Electron Microscope images [10]. In colloidal suspensions, Dynamic Light Scattering is a commonly used technique, where a laser is focused into a small volume of solution containing colloidal particles, for the determination of particle size. The size and shape of the nanoparticles determine polarization of the light scattered [11].

Another important property of nanoparticles, zeta potential, refers to the electrical potential difference between the liquid layer immediately next to the surface of a particle and the continuous phase that is further away from the particle. This value is a critical parameter indicating the surface charge of the particle. Zeta potential is measured by applying an electric field to the liquid and observing the migration of particles toward the electrodes. The stability of particles is directly related to zeta potential; high zeta potential values (positive or negative) generally mean a more stable suspension, while low values may indicate that the particles are prone to agglomeration. According to zeta potential values, systems can be defined as very unstable (± 0 –10 mV), relatively stable (± 10 –20 mV), moderately stable (± 20 –30 mV), and highly stable (± 30 mV). Therefore, optimizing zeta potential in the design and applications of nanoparticles is significant [12].

Nanoencapsulation techniques are generally classified in two approaches that are top-down and bottom-up. Top-down approaches including emulsification and emulsification-solvent evaporation and bottom-up approaches coacervation, inclusion complexation, nanoprecipitation, and supercritical fluid technique are all able to generate capsules at nanoscale in the range of 10 and 1000 nm [7]. Emulsification and solvent evaporation technique are one of the commonly used nanoencapsulation techniques for nanoencapsulation of bioactive compounds, and it is followed by spray or freeze drying. The solvent evaporation technique is carried out by emulsifying the polymer solution such as polylactic acid, polylactic-co-glycolic acid, polycaprolactone, cellulose acetate phthalate, and ethyl cellulose in an aqueous medium and then evaporating the solvent. This process allows the polymer to precipitate into nanoparticles, thus providing nanoencapsulation of the target compound. The properties of the nanoencapsulation obtained in this method are significantly affected by the viscosity of the phases, mixing speed, temperature, structure, and amount of the dispersant. The effectiveness of this technique depends mainly on the correct selection of appropriate emulsification and drying processes [13]. Emulsification technique is based on creating colloidal dispersions having particle size between 50 and 1000 nm and comprising two immiscible liquids. This technique presents an effective way to encapsulate oil-soluble bioactive food compounds and water-soluble functional ingredients [7]. Lipophilic active agent, for instance lycopene, beta-carotene, plant sterols, and vitamin E, can be delivered to food products by encapsulating into the oil in water emulsion (o/w). In contrast, water in oil emulsion (w/o) can be utilized for the encapsulation of water-soluble food active agents such as polyphenols [14]. Nanoencapsulation of essential oils is widely performed by forming nanoemulsions. Nanoemulsions are produced by stabilizing two immiscible liquids with surfactants and mixing. Oil-in-water (o/w) and water-in-oil (w/o) emulsions are the most used systems for this purpose. In o/w emulsion, the oil phase is stabilized by dispersing it

into water-based emulsifiers with mixing devices such as high-speed homogenizers, thus coating the oil droplets with emulsifiers and thus encapsulating them [15].

Coacervates are a method based on phase separation, where an aqueous solution transitions to a polymer-rich phase known as a coacervate and polymer-poor phase by a liquid-liquid phase separation mechanism. Simple coacervation involving one polymer can be achieved, or complex coacervation involving two or more polymer types with opposite ionic charges can be performed. Complex coacervates consisting of gum arabic and gelatin are often used to encapsulate active ingredients [14]. In inclusion complexation, encapsulation occurs through intermolecular interactions between cyclodextrin and the active molecule. The active or guest molecule can be transferred partly or completely into the cavity of the CD. Briefly, the inclusion complexation technique involves two different intermolecular interactions [16]. Flavonoids, which are active polyphenolic compounds, have very low water solubility, which limits their oral application and reduces bioavailability. Recently, cyclodextrin inclusion complexation method has been used to effectively improve the solubility and bioavailability of flavonoids [17]. The nanoprecipitation method stands out with its advantages over other methods such as simplicity, good reproducibility, not containing toxic solvents, obtaining small particle size distribution, and not requiring high energy. This method is based on the interfacial accumulation of polymers following the displacement of a semipolar solvent that is miscible with water from a lipophilic solution [18].

3. Freeze-drying technology for stability of nanoencapsulated bioactive compounds

Food science deals with the bioavailability and biocompatibility problems of bioactive compounds. These compounds can maintain their activity and provide their functional properties for a certain period by converting them in encapsulated form [19]. Encapsulation is a technique to be the guard for bioactive components in small capsules with a protective material against environmental and processing conditions. While microencapsulation is generally used to protect functional compounds from heat, moisture, oxygen, or other conditions, applications of nanoencapsulation technology to improve functional properties have increased in recent years. Nanoencapsulation offers advantages such as particle size reduction, increased surface area, increased bioactive properties, and improved protection and stability [20]. However, the limited ability to maintain their activity in a stable manner for a long time can be overcome by protecting them from environmental factors. Therefore, encapsulated bioactive compounds are converted into a stable form with freeze-drying technology.

Freeze-drying technology is one of the latest technologies among drying systems that provide the best quality products. Its only disadvantage is that it is a costly drying method and offers many advantages. This technology is especially preferred in products where the preservation of the functional and nutritional properties of the final product is of great importance [21]. Freeze drying is a process in which water passes directly from the solid phase to the vapor phase, by passing the liquid phase, and then desorbing the water. At the end of the process, a particularly preserved stable dry product is obtained. Thanks to this method, biological materials such as proteins, vaccines, bacteria, as well as high-quality food and drugs can be stabilized [22].

The freeze-drying process consists of three stages which are freezing, primary drying, and secondary drying (**Figure 2**). The first step, freezing, is an important step in which an aqueous solution or slurry is solidified. This can be achieved by immersion in liquid nitrogen, using cold plates operating at a controlled temperature, or spraying with a cold gas stream. Freezing rate affects both the rate of sublimation and the final properties of the dried product. Slow freezing can shorten the sublimation time due to the large pores formed by the sublimation of large ice crystals, while fast freezing is preferred to prevent the aggregation of particles. In the primary drying stage, the frozen solvent (ice) is sublimated. This sublimation is achieved by reducing the pressure below the triple point or by using a dry gas whose vapor pressure is reduced below the triple point. The critical parameter in this step is the collapse temperature, which determines the process limits during sublimation. The secondary drying stage focuses on desorption of unfrozen relative water or solvent. This step is like the final stage of ordinary drying processes, but the drying temperature must be higher than the freezing point of the solvent but not exceed the glass transition temperature of the matrix. Also, the vacuum level used in this step can be lower than in primary drying [23].

Drying technology is used to remove the solvent used in the production of nanoparticles and to obtain nanoscale powder products. Spray drying and fluidized bed coating are utilized for conversion of nanosuspension into nano powders. However, if the composition is susceptible to temperature or high residual moisture content, freeze drying is particularly preferred for this purpose. In the case of aggregation, the dissolution rate of the dried powder product may decrease. The expected

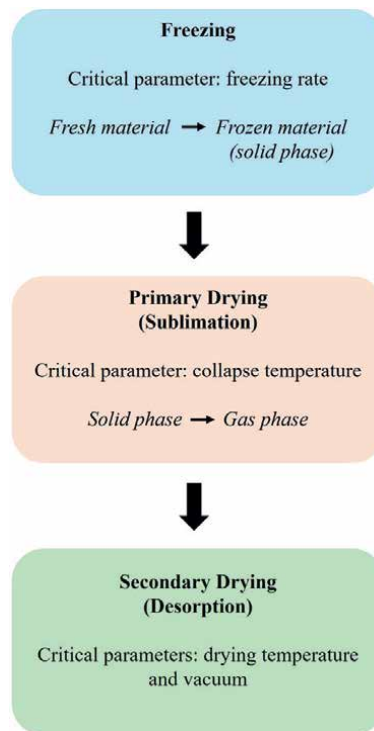


Figure 2.
The stages of freeze-drying process.

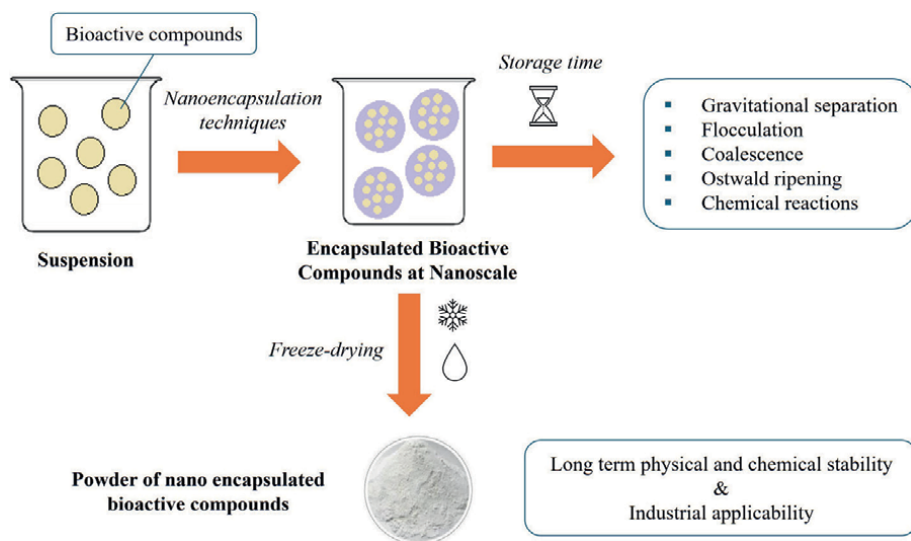


Figure 3.
The role of freeze drying in the stability of nanoencapsulated bioactive compounds.

dissolution rate and dispersibility in the final product determine the quality characteristics of the powder. Especially in pharmaceutical applications, dissolution performance may be different for each drug formulation. The process design may need to be optimized according to the target product [24]. Moreover, nanoemulsions loaded with bioactive compounds cannot maintain their stability in emulsion form for a long time. Gravitational separation, flocculation, coalescence, and Ostwald ripening phenomena that reduce kinetic stability cause this situation. These phenomena limit the ability of nanoemulsions to provide the expected functional properties during storage [25]. Although kinetic stability can be maintained with emulsifying agents in some cases, they lack thermal stability. Therefore, converting nanoemulsions into powder form provides advantages. Spray-drying and freeze-drying technologies provide high encapsulation efficiency, and nanoemulsion is transformed into an industrially applicable form as powder [26]. The advantages provided by freeze-drying technology in the production of nanoencapsulated powders of bioactive compounds are remarkable, and the method mainly plays an important role in increasing the stability of nanoformulations (**Figure 3**).

3.1 Nanoencapsulation of phenolic compounds as powder form

Phenolic compounds are among the bioactive compounds that are at the forefront in the food and pharmaceutical sectors with their many functional properties such as highly antioxidative, anti-inflammatory, and antimicrobial properties. However, due to their chemical structure, low solubility, bioavailability, and stability limit their use. Nanoencapsulation is used as an effective approach to improve these properties [27]. It is well-known that most phenolic compounds have low bioavailability. Bioavailability of phenolic compounds, which is a significant criteria to achieve the functions in the body, has been developed by nanoencapsulation [28]. Nanoencapsulation increases the bioavailability of phenolic compounds by providing

controlled release. For example, nanoencapsulation of gallic acid and quercetin in polymeric matrix provided more effective oral bioavailability [29]. When nanoparticles loaded with phenolic compounds produced by nanoencapsulation techniques are stabilized with drying technologies, they can contribute to innovative applications in both the food and pharmaceutical industries.

The powder of catechin/ β -cyclodextrin inclusion complex nanoparticles was achieved by using nanoprecipitation method followed by freeze drying. Nanoparticles having particle size lower than 100 nm were achieved by adjusting operating parameters. The stability of catechin/ β -cyclodextrin nanoparticles was evaluated based on the antioxidant activity. The antioxidant stability of catechin/ β -cyclodextrin inclusion complex nanoparticles was more than 70% after the storage period of 30 days, while the catechin was 16.42% at the end of 30 days storage. The stability of catechin was effectively maintained by nanoencapsulation and freeze drying [30]. In another study, catechin nanocapsules were developed using innovative starches obtained from three different sources including horse chestnut, water chestnut, and lotus stem. Catechin-loaded starch-based nanoparticles were produced by ultrasonication and dried in a freeze-dryer following the production process. Characterization and encapsulation efficiency analyses were performed on the obtained powder of catechin encapsulated starch-based nanoparticles. The best results in the study were obtained in horse chestnut-based starch nanoparticles, with particle size of 322.7 nm, zeta potential of -18.05 mV, and encapsulation efficiency of 59.09%. In contrast, water chestnut starch-based catechin powders showed the highest stability and slower catechin release rate in simulated gastric and intestinal digestion conditions. Nano-sized starch-based catechin powder offers bioactivity retention during *in vitro* digestion such as lipase inhibition, cholesterol esterase inhibition, DPP-IV inhibition, and glucosidase inhibition [31]. Considering the high cost of freeze-drying technology, Ruengdech and Siripatrawan [25] et al. have developed an economical alternative drying method combining the foaming method and freeze drying. In this method, the liquid sample is converted into solid foam by mixing it with foaming and stabilizing agents and then dried by the freeze-drying process. By increasing the surface area, the method provides faster drying speed and reduces the drying time, making it a more economical process. Green tea catechins encapsulated in nanoemulsions using hydroxypropyl methylcellulose as a foaming agent and maltodextrin and gum arabic as coating materials were stabilized by foam mat freeze-drying method. High product yields ranging from 87% to 91% were achieved. Encapsulation efficiency of catechins with maltodextrin and gum arabic coating materials were between 75% and 82%. The catechin nanoemulsion powder prepared with different wall materials can be used as an ingredient in the formulation of functional foods with its high antioxidant activity [25].

A variety of phenolic rich extracts were nanoencapsulated for target delivery, and freeze-drying technology plays role in the final step of production in order to maintain the bioavailability and stability of bioactives. The powder of nanoencapsulated green tea extract was produced with maltodextrin as coating material and methods of homogenization, ultrasonication treatment, and freeze drying. Particle size of freeze-dried nanoencapsulated powders ranged from 380.34 to 520.45 nm. Encapsulation efficiency of green tea polyphenols in nanocapsules was found between 41.07% and 57.53%. The developed green tea extract nanocapsules indicated controlled release in gastric and intestinal media during *in vitro* analysis [32]. In order to overcome high degradability, poor solubility and bioavailability, and intense taste, nanoencapsulation of propolis extract in sodium caseinate and maltodextrin nanocomplex was

performed by Soleimanifard, Feizy [33]. Different ratios of propolis-loaded nano-complex were produced by stirring, homogenization, and ultrasonication steps. Then, it was converted to propolis nanocomplex pellets by freeze drying. Particle size values were higher than 500 nm, which was associated with an increase in the number of larger molecules of sodium caseinate. Spherical and smooth structures were observed in morphology of nanocomplexes. Encapsulation efficiencies of propolis extract were changing from 67.48% to 96.18% [33].

Literature studies clearly reveal that fruit juice industry waste can be converted into value-added components with functional properties thanks to technologies of nanotechnology and freeze drying. The other study investigated nanoencapsulation of pomegranate peel extract using maltodextrin and whey protein. Similarly, the production steps included homogenization to make composition homogenous, ultrasonication treatment to decrease the particle size at nanoscale and freeze drying to obtain final product in powder form. It was achieved in the study that encapsulation efficiency of pomegranate peel extract was between 85% and 96%. In addition, antiproliferative activity of nanoencapsulated pomegranate peel extract demonstrated its inhibition on the growth of cancer cells. Thus, many functional phytochemicals in the pomegranate peel extract were encapsulated and protected by using nanotechnology and freeze drying [34]. Passion fruit waste, another fruit juice industry waste, is a potential source of bioactive compounds. Poly (dl-lactide-co-glycolide) (PLGA)-based nanoparticles loaded with passion fruit by-products extracts were produced by solvent evaporation technique to improve the bioactivity of extract as well maintain controlled release. PLGA nanoparticles encapsulated passion fruit by-products extracts had particle size and encapsulation efficiency in the range of 355 to 470 nm and 24–79%, respectively. Controlled release of the nanoparticles was investigated in the phosphate buffered saline, and results noticed that 72–88% of the extract was released from the nanoparticles within 24 hours and remained at this level up to 72 hours. The study pointed out the opportunities to utilize passion fruit waste through nanotechnology and freeze-drying applications [35].

3.2 Nanoencapsulation of essential oils as powder form

Due to the increasing awareness of the benefits of functional foods, studies on the development of new functional food products using the bioactive compounds are encouraged. Plant essential oils are widely used to increase functional properties of foods. However, they are very sensitive to harsh conditions such as oxidation and high temperatures. To overcome these limitations, nanoencapsulation applications, one of the latest technologies for incorporating essential oils into different systems (food or food packaging), have been developed. Nanoencapsulation increases the stability of essential oils, provides controlled release, and improves their functional activities, especially their antioxidant properties [15].

Essential oils encapsulated by creating nanoemulsions can be converted into stable powder products by freeze drying. Foam mat freeze drying, an improved more economical freeze-drying technique, was used to stabilize the nanoemulsion encapsulating clove essential oil. The clove essential oil is well-known for its antimicrobial activity and is utilized as food additive. Its direct use is restricted by its strong flavor and poor water solubility. With nanoemulsion technology, these limitations for clove essential oil can be overcome. Nanoemulsion of clove essential oil was prepared by ultrasonication and dried to obtain stable product by foam freeze drying. Foam freeze-dried nanoencapsulated clove oil with a particle size of 26.14 nm and a

solubility of 88.71% was developed under optimized production conditions. In addition, its antimicrobial activity against gram-negative *Escherichia coli*, *Pseudomonas aeruginosa*, and gram-positive *Staphylococcus aureus* showed high stability during 3 months storage at 25°C [36]. In another study, the best *Artemisia absinthium* L. essential oil obtained by ultrasound-assisted extraction was encapsulated by loading into maltodextrin-based nanoparticles. Nanoencapsulated powder of *Artemisia absinthium* L. essential oil was manufactured by freeze drying. Bioactive compounds particularly phenolics in the *Artemisia absinthium* L. essential oil are vulnerable to oxidation in the presence of moisture, oxygen, and light. Therefore, such compounds including unsaturated bonds in the molecular structure require encapsulation for usage in food products with protected functional properties. An increase in the ratio of maltodextrin as a coating material resulted in increasing encapsulation efficiencies changing from 75.21% to 85.24%. A low degree of crystallinity was revealed by XRD measurements [37].

Extracts and essential oils of some medicinal plants have an important place as functional compounds in the human body, foods, and packaging materials due to their considerable antioxidant and antimicrobial properties. Chavir (*Ferulago angulata*) has widely been used in dairy products for antimicrobial purposes. Despite their rich functional properties, the high amount of flavoring substances in the composition of essential oils limits their use in foods. For this purpose, the Chavir (*Ferulago angulata*) essential oil was encapsulated in chitosan nanoparticles using the ionic gelation method and freeze drying. Maximum encapsulation efficiency and loading capacity was determined as 60.20% and 24.5%, respectively. Particle size of nanoparticles encapsulated essential oil was in the range of 175 and 350 nm. Spherical structure of particles was proved by SEM images [38]. Quinoa oil, rich in linoleic and α -linolenic unsaturated fatty acids, has been nanoencapsulated to enhance its functional properties and protect fatty acids from oxidation. Nanoemulsion technique was used to produce nanoencapsulated quinoa oil with whey protein isolate and gelatine as encapsulating agents. Then, the freeze-drying process was applied to the produced nanoemulsion. Characteristics of the nanoparticles indicated that water dispersibility was advanced as the value higher than 70%, and the antioxidant activity of quinoa oil was improved. Particle size of nanoencapsulated quinoa oil ranged from 161 to 264 nm. Encapsulation efficiency of nanoparticles was found at a high level between 74% and 83% [39]. Lemon essential oil is rich in bioactive compounds and is sensitive to light and oxidation. In the study, chitosan and modified starch-based nanocapsules in which lemon essential oil was encapsulated were obtained in stable form by freeze-drying technology while preserving the product properties. The highest encapsulation efficiency achieved in nanocapsules was 85.44%, and the minimum particle size of nanocapsules was 339.3 nm, while greatest value reached 553.3 nm. The release kinetics of oil from nanocapsules pointed out an early spread release followed by gradual release [40].

3.3 Nanoencapsulation of oils rich in polyunsaturated fatty acids as powder form

Foods rich in polyunsaturated fatty acids (PUFAs) are important in terms of human nutrition to consume adequate amount of essential fatty acids. Fish oil, which is rich in polyunsaturated fatty acids, is the most well-known omega-3 source with the composition of eicosapentaenoic acid and docosahexaenoic acid. In addition, plant

based oils such as flaxseed oil, walnut oil, and chia seed oil are rich in polyunsaturated fatty acids [6]. However, polyunsaturated fatty acids are not provided to the body in sufficient amounts due to their tendency to deteriorate during processing or storage and their poor stability, solubility, and absorption properties in the human intestine. Therefore, nanoscale delivery systems are being developed to improve the bioavailability, stability, and maintain the efficacy of bioactive lipids rich in polyunsaturated fatty acids [41].

A bilayer nano-oleogel was formed by encapsulating fish oil with different proportions of glyceryl monostearate, soy lecithin, and whey protein isolate. In the production of nano-oleogel, freeze-drying technique was used to remove water after obtaining oleogel/water nanoemulsion. In terms of the particle size, the first layer coating with lecithin resulted in 144.8 nm, while adding the second layer coating with whey protein isolate raised to 561.5 nm. Food application was performed on cookies by using fish oil oleogel for commercial shortening in baking with a 100% substitution rate. The appearance properties did not show any significant difference between cookies prepared with oleogel and those prepared with commercial shortening. Only cookies made with double-coated nano-oleogel a slightly darker color, probably due to WPI denaturation by heating [42]. In another study, krill oil at different concentrations was encapsulated in whey protein concentrate, maltodextrin, and gum arabic formulations using the nanoemulsion method. Following this, the powder nanocapsules were obtained as a result of the spray drying and freeze-drying processes and the properties of nanoencapsulated krill oil powder were compared. The highest encapsulation efficiency was determined as 72% in the spray dried sample and 67% in the freeze-dried sample in the formulation containing 8% (w/v) krill oil. In terms of oxidative stability, the samples obtained with both drying methods showed similar trends during storage. *In vitro* bioavailability ranged from 32.7% to 72.4% in freeze-dried nanoencapsulated krill oil powder, while it ranged from 20.0% to 83.0% in spray-dried nanoencapsulated krill oil powder [26].

In order to limit lipid oxidation of high amount of PUFAs in flaxseed oil, nanocapsules were developed by encapsulating flaxseed oil with the method of homogenization, ultrasonication, and freeze-drying techniques. The noteworthy point in the study is the use of flaxseed gum, a by-product of flaxseed oil preparation, in the encapsulation of flaxseed oil. The developed flaxseed oil nanocapsules having 208.22 nm particle size indicated considerably high encapsulation efficiency (96%). The zeta value determined as -21.98 mV showed that the particles had high stability. Nanoencapsulated flaxseed oil showed higher oxidative stability over 7 days than nonencapsulated flaxseed oil. Flaxseed oil nanocapsules developed in the study proved that their oxidative stability was improved by determining the fatty acid composition, peroxide value, and oxidative stability index [43]. In another study, chia seed oil was similarly nanoencapsulated using chia seed mucilage. Nanoparticles with spherical shapes and an average size of 205 nm were obtained. Encapsulation efficiency was determined as 82.8%, loading capacity as 35.38%. FT-IR analysis confirmed the interaction between oil and mucilage, while thermal analysis showed that the nanocapsules were stable up to 300°C. Accelerated storage tests for 28 days revealed that the nanoencapsulated chia seed oil was more resistant to oxidation. The results also indicate that chia seed mucilage may be a natural alternative to synthetic polymers. **Table 1** summarizes these recent studies on the nanoencapsulation of bioactive compounds including phenolic compounds and phenolic rich extracts, essential oils, and oils rich in PUFAs by using freeze-drying technology [44].

	Bioactive ingredient	Production methods	Final product	Main results	Ref.
Phenolic compounds/ phenolic rich extracts	Catechin	Nanoprecipitation and freeze drying	Powder of catechin/ β -cyclodextrin inclusion complex nanoparticles	Particle size < 100 nm, high antioxidant stability > 70% after 30 days of storage	[30]
	Catechin	Ultrasonication and freeze drying	Powder of catechin encapsulated starch-based nanoparticles.	322.7 nm particle size, 59.09% encapsulation efficiency, bioactivity retention during <i>in vitro</i> digestion	[31]
	Green tea catechins	Nanoemulsion formation and foam mat freeze drying	Powder of green tea catechin nanoemulsion	87–91% product yields, 75–82% encapsulation efficiency, high antioxidant activity	[25]
	Green tea extract	Homogenization, ultrasonication, and freeze drying	Powder of nanoencapsulated green tea extract	380.34–520.45 nm particle size, 41.07–57.53% encapsulation efficiency, controlled release during <i>in vitro</i> digestion	[32]
	Propolis extract	Homogenization, ultrasonication, and freeze drying	Powder of propolis-loaded nanocomplex	particle size > 500 nm, 67.48–96.18% encapsulation efficiency	[33]
Essential oils	Pomegranate peel extract	Ultrasonication and freeze drying	Powder of nanoencapsulated pomegranate peel extract	85–96% encapsulation efficiency, antiproliferative activity against cancer cells	[34]
	Passion fruit by-products extracts	Emulsion solvent evaporation technique and freeze drying	Powder of nanoencapsulated passion fruit by-products extracts	355–470 nm particle size, 24–79% encapsulation efficiency, controlled release in phosphate buffered saline	[35]
	Clove essential oil	Nanoemulsion formation and freeze drying	Powder of nanoencapsulated clove essential oil	26.14 nm particle size and 88.71% solubility, antimicrobial activity stability during 3 months storage at 25°C	[36]
	<i>Artemisia absinthium</i> L. essential oil	Homogenization and freeze drying	Powder of nanoencapsulated <i>Artemisia absinthium</i> L. essential oil	75.21–85.24%, encapsulation efficiencies, a low degree of crystallinity	[37]
	Chavir (<i>Ferulago angulata</i>) essential oil	Ionic gelation method and freeze drying	Powder of nanoencapsulated Chavir essential oil	175–350 nm particle size, 60.20% maximum encapsulation efficiency, 24.5% loading capacity	[38]
	Quinoa oil	Nanoemulsion formation and freeze drying	Powder of nanoencapsulated Quinoa oil	161–264 nm particle size, 74–83% encapsulation efficiency, advance water dispersibility (>70%) and antioxidant activity	[39]
	Lemon essential oil	Homogenization, ultrasonication, and freeze drying	Powder of nanoencapsulated lemon essential oil	339.3–553.3 nm particle size, 85.44% the highest encapsulation efficiency early spread release followed by gradual release of oil	[40]

Bioactive ingredient	Production methods	Final product	Main results	Ref.
Oils rich in PUFAs	Fish oil	Oleogel/water nanoemulsion and freeze drying	Nano-oleogel powder encapsulating fish oil with double layer and cookies formulated with nano-oleogel fish oil powder	[42]
	Krill oil	Nanoemulsion and spray/freeze drying	Powder of krill oil nanocapsules	
	Flaxseed oil	Homogenization, ultrasonication, and freeze drying	Powder of flaxseed oil nanocapsules	
	Chia seed oil	Homogenization and freeze drying	Powder of chia seed oil nanocapsules	
			For spray dried and freeze-dried sample: 72% and 67% encapsulation efficiency, 20–83% and 32.7–72.4% <i>in vitro</i> bioavailability	[26]
			208.22 nm particle size, 96% encapsulation efficiency, high oxidative stability over 7 days	[43]
			205 nm particle size, 82.8% encapsulation efficiency, 35.38% loading capacity, oxidative stability	[44]

Table 1.
Nanoencapsulated bioactive ingredients, production methods, and main results.

4. Future perspective

Nanoencapsulation is applied in many areas, especially in medicine, food, and biotechnology. Freeze-drying technology is of great importance in preserving the active ingredients of nanocapsules and thus extending their shelf life and maintaining product quality. However, when considering scale-up and industrial application, some difficulties are encountered. Since freeze drying is a high-cost process, the high energy required when the scale is increased causes high costs for the product. Additionally, the long process time including pretreatments and lyophilization also limits the production speed. For this, the process can be designed to minimize energy consumption. For instance, appropriate temperature and pressure conditions can be determined during the pre-freezing stage to shorten the drying time. In addition, the drying time can be reduced by increasing the dry matter content. Alternatively, energy efficiency can be increased by optimizing the vacuum level.

Despite the difficulties, when the profit and loss balance is considered, these limitations can be overlooked when the final product is a functional food or drug. The product appealing to a certain consumer group can be offered for sale in certain quantities and at a high price. In order to increase the applicability of freeze-drying technology, which can produce promising nanocapsule products for the future, it is necessary to use new cryoprotectants and stabilizers suggested by researchers and to optimize the conditions to shorten the production period.

5. Conclusion

Nanoencapsulation is one of the encapsulation technologies that have become widespread in recent years. Bioactive compounds can be nanoencapsulated for different purposes. Some of these purposes are to protect against deterioration accelerating factors such as oxidation, light, and moisture, to provide controlled release, to increase water solubility, and to further improve antioxidant and antimicrobial properties. Freeze-drying technology is used to transform nanocapsules that provide these properties into a stable form and to ensure commercial applicability. In this book section, information is given about nanotechnology methods used in the encapsulation of bioactive compounds and the characteristic properties of nanocapsules. Following this, freeze-drying technology and its role in converting nanoencapsulated products into powder form are summarized. Current literature studies in which nanoencapsulation of phenolic compounds, essential oils, and polyunsaturated fatty acids is performed and powder form products are developed with freeze-drying technology are presented. This information will shed light on innovative studies planned.

Conflict of interest

The author declares no conflict of interest.

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Infrared Drying in Food Technology: Principles and Practical Insights

Hassan Sabbaghi and Phuong Nhu Nguyen

Abstract

Infrared drying marks a revolutionary advancement in food processing, offering significant improvements in both operational efficiency and product integrity. By utilizing specific wavelengths of radiation, this approach achieves rapid, uniform heat transfer and effective moisture removal across a diverse range of food products. Its versatility spans applications in grains, fruits, vegetables, meats, seafood, and snack foods, adapting to the distinct requirements of each category. In addition to preserving the nutritional profile of food, this technique enhances rehydration properties and sensory attributes, ensuring superior consumer acceptance. It provides precise control over processing conditions, which helps retain texture, flavor, and bioactive compounds, even for delicate products. Practical insights underscore this method's ability to reduce processing times and improve energy use, leading to cost-effective operations without compromising output quality. As an advanced drying technique, it surpasses traditional methods by addressing modern food production challenges. Compliance with regulatory standards ensures food safety and consistent quality, meeting stringent requirements. Infrared drying serves as a foundation for developing hybrid technologies, offering advanced strategies for specialized processing needs. This transformative innovation is poised to shape the future of food manufacturing, enabling progress while meeting evolving consumer and technological demands.

Keywords: infrared drying, product integrity, radiation, practical insights, food industry

1. Introduction

Drying is a fundamental process in the food industry, designed to preserve food by reducing its moisture content, thereby inhibiting microbial growth and enzymatic reactions. Traditional drying methods, such as sun drying, are cost-effective but have significant limitations, including dependency on weather conditions and risks of contamination. Industrial methods like hot air drying address some of these issues but often lead to uneven drying, loss of nutritional quality, and high-energy consumption.

With advancements in food technology, modern drying methods, such as freeze-drying and microwave drying, have offered solutions but come with their own challenges of cost and complexity [1]. Thus, Infrared (IR) drying has emerged as an innovative alternative, bridging the gap between traditional and advanced techniques. By utilizing electromagnetic radiation within the infrared spectrum (0.78–1000 μm), this method offers direct and efficient heat transfer, resulting in rapid moisture evaporation [2]. Unlike convection-based methods, where heat is transferred through surrounding air, infrared drying directly excites water molecules in food, ensuring energy efficiency and shorter drying times. These characteristics make it particularly relevant for the food industry, where the balance between energy use, time, and product quality is critical [3].

Infrared drying stands out for its precision, allowing targeted applications across various food categories, including grains, fruits, vegetables, and meats. Its ability to preserve sensory and nutritional qualities, such as color, flavor, and vitamins, while ensuring microbial safety, makes it particularly effective for high-value products [4]. Additionally, its alignment with sustainability goals, such as reducing energy consumption and minimizing environmental impact, further enhances its relevance in modern food processing [5, 6]. Technological advancements, including hybrid systems combining infrared with vacuum or microwave drying, have improved drying efficiency and broadened its applications. Automation and monitoring systems have enhanced the reliability and scalability of infrared drying, making it suitable for both small-scale and industrial operations. These innovations underscore its transformative potential for the food industry [7, 8].

The present chapter provides an overview of infrared drying principles, recent technological advancements, and diverse applications in food processing. It offers practical insights into equipment design, operational parameters, and optimization for various products. Environmental and economic benefits, regulatory and safety considerations, and future opportunities for industrial adoption and innovation are also discussed.

2. Principles of infrared drying

2.1 Definition and basic principles

Infrared radiation occupies the spectrum between 0.78 and 1000 μm , which is divided into three regions based on the electromagnetic spectrum shown in **Figure 1**. Near Infrared (NIR, 0.78–2.5 or 3 μm), Mid Infrared (MIR, 2.5 or 3–25 or 30 μm), and Far Infrared (FIR, 25 or 30–1000 μm). Each region exhibits unique energy characteristics suited to different thermal applications. NIR penetrates deeply into materials with high energy, MIR offers a balance between surface and depth heating, and FIR provides low-energy, surface-focused heating for gentle processes. Infrared wavelengths ranging from 2.5 to 200 μm are predominantly utilized for drying applications [9, 10].

2.2 Mechanism of heat transfer and moisture removal

The physics of infrared radiation forms the foundation for its practical applications in controlled energy transfer and rapid heat generation. The mechanism of infrared drying involves the direct absorption of infrared energy by the food's

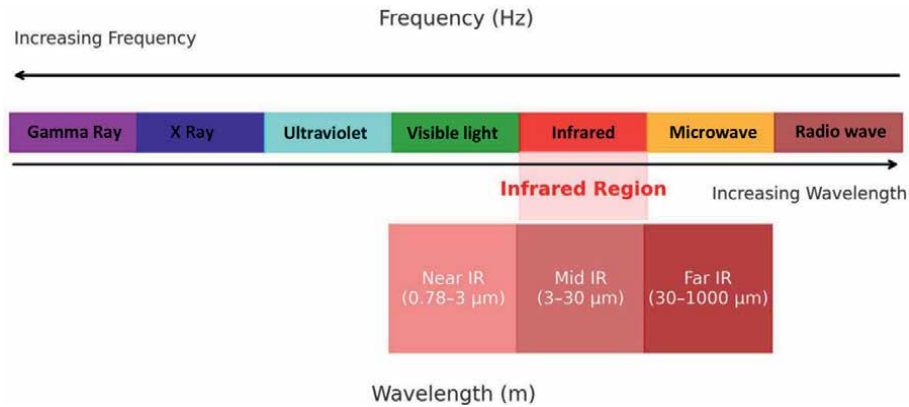


Figure 1.
Infrared region within the electromagnetic spectrum: Near, Mid, and Far IR.

surface, which then penetrates into the material. Infrared energy excites water molecules, causing them to vibrate and generate localized heat. This heat promotes rapid evaporation of moisture from the surface, creating an efficient drying process. The penetration depth of infrared energy depends on the radiation wavelength: shorter wavelengths penetrate deeper into the material, facilitating internal moisture removal, while longer wavelengths are ideal for surface heating. Water exhibits significant absorption of infrared radiation at wavelengths approximately 2.7–3.3, 6.0, and beyond 12.5 μm. The absorption of IR energy by the O-H bonds in water molecules induces rotational motion at frequencies matching the incident radiation, effectively converting IR energy into rotational energy, which drives water evaporation. When infrared radiation interacts with a surface, it can be reflected (ρ), absorbed (α), or transmitted (τ). If the material has minimal transmissivity, the IR radiation is either reflected or absorbed, based on the radiation properties and the surface characteristics. This behavior is defined by emissivity (ϵ), a value ranging from 0 to 1. Materials acting as perfect blackbodies fully absorb incoming radiation and exhibit an emissivity of 1.0, while entirely reflective surfaces have an emissivity of 0 [10, 11]. The process by which infrared waves travel from their source and interact with semitransparent materials is conceptually shown in **Figure 2**.

The efficiency of infrared drying is influenced by the physical and chemical characteristics of the food matrix, including moisture content, porosity, and thermal properties.

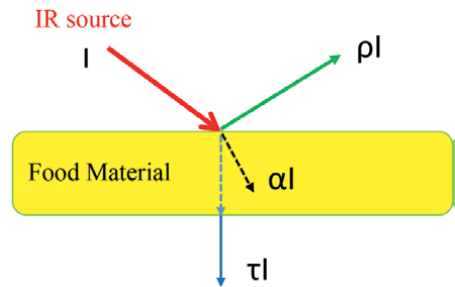


Figure 2.
Illustration of infrared (IR source) radiation interactions with a semitransparent material: Incident radiation (I), Reflected + Emitted (ρI), Absorbed (αI), and Transmitted (τI).

High-moisture foods absorb energy more effectively, leading to faster drying rates. Porous structures enable improved vapor diffusion, accelerating moisture removal [12]. Furthermore, tailored wavelengths and controlled heating cycles help achieve uniform heat distribution, reducing the risk of overheating and quality degradation. This method ensures consistent drying, even in materials with complex geometries or variable compositions. Unlike conventional drying techniques, where uneven heat transfer can lead to localized over- or under-drying, infrared drying minimizes these issues. The ability to penetrate the food matrix with precise energy application enhances structural integrity and preserves sensory and nutritional qualities. Infrared drying also aligns with energy efficiency goals, requiring less overall energy compared to convection-based drying methods. By focusing energy directly on the material, it eliminates heat loss to the surrounding air, reducing operational costs and environmental impact [13].

2.3 Comparison with other drying methods

Infrared drying distinguishes itself from other methods by combining energy efficiency with high precision, making it an essential tool for process optimization and hybrid system design. By utilizing electromagnetic radiation, infrared drying achieves rapid and uniform heat transfer, addressing the limitations of traditional techniques. A closer examination of hot air, microwave, and vacuum drying reveals the unique benefits of infrared drying in improving process outcomes [14, 15]. *Hot air drying*, a conventional method, relies on convective heat transfer through surrounding air. This indirect mechanism often results in uneven heating and prolonged drying times, increasing the risk of over-drying and nutrient loss. Infrared drying overcomes these challenges by delivering targeted, direct energy to the material's surface, ensuring uniform heating and retaining nutritional and sensory qualities, particularly in products sensitive to heat [16–18]. *Microwave drying* generates heat internally through electromagnetic waves, achieving shorter drying times compared to hot air methods. However, its tendency to create hotspots and uneven moisture distribution can compromise product quality. Infrared drying mitigates these drawbacks by offering a controlled and consistent heat application, allowing uniform moisture removal while maintaining structural integrity. For delicate products like fruits and vegetables, infrared methods demonstrate superior performance in preserving texture and color compared to microwave drying [19–21]. *Vacuum drying* operates under reduced pressure to lower the boiling point of water, enabling gentle drying for heat-sensitive products. Although effective, this method requires longer processing times and higher energy consumption. Infrared drying, when integrated into vacuum systems, addresses these issues by accelerating drying rates and enhancing energy efficiency. This hybrid approach is particularly valuable for processing fragile materials, such as herbs and teas, where quality retention is critical [22–24].

As shown in **Table 1**, infrared drying stands out across key parameters, offering a unique combination of speed, energy efficiency, uniformity, and product quality. Its adaptability for standalone use and integration into hybrid systems makes it a powerful tool for improving food processing techniques and achieving superior results across various applications. Infrared drying offers high nutritional and sensory retention due to its uniform heat application and precise moisture control. Hot air and microwave drying methods provide moderate quality preservation but are prone to overheating; hot air drying often leads to gradual overheating during prolonged exposure, while microwave drying can cause localized overheating due to uneven

Drying method	Drying time	Energy efficiency	Uniformity	Product quality	References
Infrared	Short	High	Excellent	High	[14–16]
Hot air	Long	Moderate	Variable	Moderate	[16–18]
Microwave	Very short	High	Poor	Variable	[19–21]
Vacuum	Moderate	Moderate	Good	High	[22–24]

Table 1.
Comparison of infrared drying with other methods.

energy distribution. In contrast, vacuum drying ensures high product quality through its gentle drying process, making it ideal for sensitive foods [14–23, 25].

3. Infrared drying equipment and technologies

3.1 Types of infrared dryers

Infrared dryers are classified into three main types: *batch dryers*, *continuous dryers*, and *hybrid systems*, each tailored to meet specific operational needs. Batch dryers are designed for small-scale operations, where precise control of drying conditions is essential. These systems are particularly suited for high-value or experimental products that require specific moisture levels and consistent quality. On the other hand, continuous dryers are employed in large-scale industrial processes, providing a steady flow of materials through the drying chamber. Their design ensures uniform heat application and efficient handling of high-throughput production lines. Hybrid infrared systems, which combine infrared with other drying methods such as hot air or vacuum drying, offer enhanced drying performance by leveraging the strengths of multiple technologies [26–29].

3.2 Various emitters

Infrared emitters are classified by wavelength into short-wave (SWIR), medium-wave (MWIR), and long-wave (LWIR) types. Short-wave (SWIR) emitters, such as metallic elements or incandescent lamps, produce high-intensity heat, ideal for rapid drying applications like grains and dense product but may risk overheating. Medium-wave (MWIR) emitters, often made from quartz, balance penetration and surface heating, making them suitable for drying fruits and vegetables like apples and carrots. Long-wave (LWIR) emitters, commonly ceramic-based, provide gentle surface heating, ideal for preserving delicate products such as herbs without causing thermal degradation. These material-based designs align with specific food processing needs [30, 31]. The comparison of different types of infrared emitters based on their wavelength is summarized in **Table 2**.

Emitters are further divided into electrical and gaseous types. Electrical emitters operate at 1100–2200°C, achieving high efficiency (78–85%) and radiant intensities up to 400 kW/m². They are versatile and provide precise energy control but are costly. Gaseous emitters operate at 343–1100°C, offering lower costs and durability, with radiant intensities up to 22 kW/m². Despite lower conversion efficiency (40–46%), they are popular in industrial applications due to their reliability [3, 32]. Electrical infrared emitters include reflector emitters, incandescent lamps, quartz tubes, and ceramic

Emitter type	Short-wave (SWIR)	Medium-wave (MWIR)	Long-wave (LWIR)
Wavelength	< 1.4 μm	1.4–3.0 μm	> 3.0 μm
Emitter sub-Type	Incandescent Lamps, Metallic Elements	Quartz Tube	Ceramic Element
Heat Output	High	Moderate	Moderate
Advantages	High intensity, deep material penetration	Fast heat-up, precise thermal control, prevents quality loss	Durable, resistant to thermal shocks, Gentle heating
Disadvantages	Overheating risks	Fragile, sensitive to impact, Requires precise control	Lower penetration depth
Applications	Rapid drying of grains and dense products	Continuous drying of fruits and vegetables	Drying herbs and delicate foods
Suitability for Food Drying	Moderate	High	High
References	[10, 29–32]	[10, 29–33]	[10, 29–34]

Table 2.
Characteristics and applications of infrared emitters based on wavelength.

elements. These emitters use heated metal filaments to generate radiation, offering flexibility in energy levels and modular configurations for customized intensity. Ceramic and quartz emitters are commonly used in drying systems, with standardized power values for industrial needs. Gaseous emitters are categorized into direct flame and catalytic flameless types. Direct flame emitters rely on combustion, often wasting energy due to visible light and posing safety risks. Catalytic emitters, emitting mid- and far-infrared radiation without visible flames, are more efficient and environmentally friendly. These emitters are preferred for their consistent performance and adaptability across various applications. Both types of emitters demonstrate varying penetration depths and efficiency based on product characteristics and spectral peaks [3, 30–34]. Catalytic IR emitters, for instance, can maintain surface temperatures near 500°C, generating a peak wavelength of 3.7 μm , ideal for food drying [35].

3.3 Design and operational parameters

The performance of infrared dryers is significantly influenced by their design and operational parameters. Key factors include *emitter power, distance between the emitter and the product, thickness of the product, and drying time*. Higher emitter power reduces drying time but increases the risk of overheating and quality degradation if not properly managed. Conversely, adjusting the emitter distance can optimize uniform heat distribution, which is crucial for delicate products like fruits and vegetables [30, 36, 37]. An increase in sample thickness significantly prolonged drying time, with its impact being more substantial than that of the distance from the infrared heater [36, 38]. Modeling of mass transfer revealed a notable increase in the effective diffusion coefficient with rising surface temperature and thickness, with higher activation energy for thinner samples, indicating a pronounced dependence on irradiation temperature as thickness decreased [38]. A measurement system was developed to evaluate the radiant energy efficiency and heat flux distribution of infrared heaters, revealing that efficiency is strongly influenced by input power and, for in-plane efficiencies, by heater distance [39]. *Airflow within the drying chamber* is

a particularly critical parameter in IR drying. Effective airflow facilitates the removal of evaporated moisture, preventing its accumulation near the product surface, which could otherwise hinder drying efficiency. Infrared systems, when integrated with controlled airflow, enhance heat transfer by maintaining uniform temperature and humidity levels across the drying chamber. This is especially advantageous for thin-layer products, where IR radiation efficiently combines with airflow to achieve rapid and uniform moisture removal. Moreover, optimizing drying time in IR systems ensures that thermal energy is utilized effectively, minimizing energy consumption while preserving product quality [40, 41]. Effective design and operational control contribute to the scalability and versatility of IR drying systems, as demonstrated in industrial applications for foods [42, 43].

Table 3 provides a concise summary of the effects of varying infrared drying parameters, including power, distance, time, thickness, and airflow rate, on drying rate and their industrial relevance in diverse applications.

3.4 Advances in infrared drying technology

Infrared heaters have seen significant advancements in design and functionality, as detailed in the referenced article. Innovations in the structural design of heaters,

Parameter	Status	Impact	Industrial relevance	Reference
Power	High	Significantly increases speed; risks overheating	High-throughput operations like grains	[30, 31, 36]
	Low	Slows drying; ensures gentle heating	Sensitive products like herbs and leafy greens	
Distance	Short	Speeds up drying; risks uneven heating	Dense products requiring fast drying, e.g., meats	[30, 36, 37]
	Long	Slows drying; improves uniformity	Uniform drying for fruits and vegetables	
Time	Extended	Ensures complete drying; slower process	Premium applications ensuring quality, e.g., dried fruits	[30, 36, 40]
	Reduced	Speeds up drying; risks incomplete removal	Time-sensitive snacks and ready-to-eat meals	
Thickness	Thin <2 mm	Shorter drying times; rapid moisture removal	Suitable for delicate products requiring minimal exposure	[30, 36–38]
	Moderate 2–5 mm	Balanced drying rate; potential for uniform heat penetration	Ideal for most food products ensuring quality retention	
	Thick >5 mm	Prolonged drying times; risk of uneven heating	Best for robust items with adjusted drying conditions to prevent overheating	
Airflow	High	Enhances speed by removing moisture quickly	Improves efficiency in seafood and high-moisture items	[36, 40–43]
	Low	Slows drying due to humidity buildup	Precise drying where gentleness is critical	

Table 3.
Effects of design parameter adjustments on infrared drying and industrial significance.

including modular configurations, allow for customizable heat distribution suited to various industrial needs. Integration with smart control systems further enables precise regulation of heat output, adapting to specific processes and reducing energy consumption. These advancements collectively enhance the performance and reliability of infrared heaters in industrial applications, including drying and heating processes [30, 39].

Hybrid drying systems, often referred to as *IR-assisted drying*, integrate infrared (IR) radiation with complementary drying methods to enhance both efficiency and product quality. The term “IR-assisted” highlights the pivotal role of infrared radiation in these configurations, leveraging its ability to deliver rapid surface heating while the secondary methods address deeper moisture removal. This synergy accelerates drying rates and optimizes the preservation of sensitive nutritional and sensory properties. For instance, IR-assisted drying is particularly beneficial for fruits, minimizing vitamin degradation and maintaining vibrant colors, while in grains and cereals, it ensures uniform moisture removal, enhancing cooking properties and shelf life. Common examples include IR-assisted hot air drying, microwave drying, and freeze-drying, each tailored to specific industrial applications and product requirements [4, 36, 44]. IR-assisted hot air drying consists of an infrared radiator, a hot air circulation system, and a control unit for temperature and airflow regulation [40]. A typical vacuum infrared (IR) drying system comprises an insulated chamber capable of withstanding low-pressure conditions, an IR radiator or heater to deliver thermal radiation to the product, a vacuum pump to sustain the vacuum within the chamber, and a control system for regulating the IR radiator. **Figure 3** illustrates the schematic layout of a vacuum IR dryer [45]. IR-assisted freeze-drying combines these principles with sublimation at low temperatures, ensuring efficient drying while preserving sensitive product qualities [46].

In certain cases, the combination of far-infrared radiation and low-pressure superheated steam enhances heat transfer efficiency while preserving product quality by reducing thermal degradation. This setup is illustrated in **Figure 4**. The far-IR radiator was controlled based on the temperature measured above the sample surface. Drying experiments were conducted at 70, 80, and 90°C with chamber pressures of 7 and 10 kPa [47].

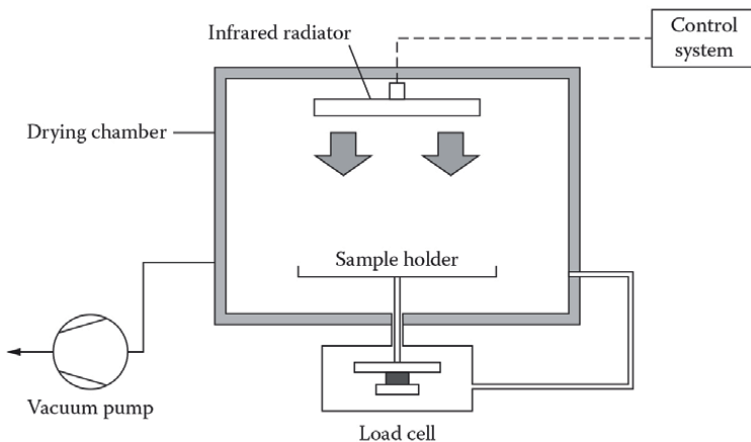


Figure 3.
A diagram illustrating the schematic design of a vacuum IR dryer.

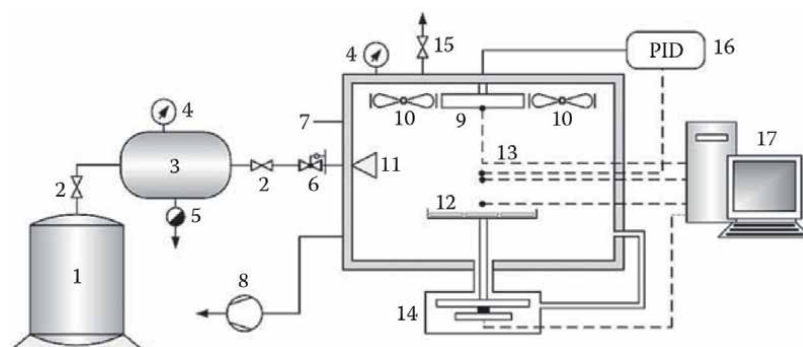


Figure 4. Diagram of a combined far-infrared radiation and low-pressure superheated steam drying apparatus. Key components include: (1) Steam generator, (2) Control valve for steam flow, (3) Steam buffer tank, (4) Pressure monitoring gauge, (5) Condensate separator or steam trap, (6) Adjustable steam regulator, (7) Insulated drying chamber, (8) Vacuum pump, (9) Far-infrared radiator, (10) Far-infrared emitter, (10) Cooling and circulation fans, (11) Steam distributor and inlet system, (12) Sample holder, (13) Thermocouples for temperature measurements, (14) Load cell for weight assessment, (15) Vacuum breakup valve, (16) Proportional-Integral-Derivative (PID) controller for system regulation, and (17) Personal computer (PC) with data acquisition interface.

Advancements in *compact system* designs have expanded the flexibility of infrared drying systems, allowing seamless integration into existing production lines. These modular configurations are particularly advantageous for snack foods and baked goods, where scalability and consistent quality are essential. Compact designs also reduce the overall footprint of drying systems, making them more accessible for small- and medium-scale food processors [48]. Infrared drying can be designed as a *simultaneous process*, integrating blanching, disinfestation, and drying in a single step. This approach enhances efficiency by combining multiple operations, reducing processing time, and maintaining product quality through precise control of heat and moisture [49, 50]. In simultaneous systems, infrared heating in *continuous method* (constant power) provides consistent energy delivery, leading to uniform heating but may risk overheating sensitive products. In contrast, the *intermittent method* (constant surface temperature) adjusts power delivery based on product surface conditions, optimizing energy efficiency and preventing thermal degradation [51]. Simultaneous IR drying and disinfestation offers a rapid, nonchemical method to eliminate insect pests in grains while preserving product quality. The system, illustrated in **Figure 5**, utilizes a catalytic IR emitter and a vibration bed with adjustable speed, ensuring even heat distribution for effective pest control and quality retention [50].

Another transformative advancement is the integration of *automation technologies*, which have revolutionized infrared drying processes. Modern systems now include real-time monitoring and advanced sensors that dynamically adjust key parameters, such as temperature, emitter intensity, and humidity levels. For example, automated systems effectively manage intermittent heating cycles in infrared drying, reducing thermal stress and preserving product quality. These systems have demonstrated particular benefits in applications such as the drying of fruit slices using a *step-cooking* approach, where fuzzy logic controllers ensure consistent surface temperature regulation and optimal moisture removal. By minimizing over-drying and under-drying, automation supports sustainable production practices while maintaining high-quality standards in processed foods. Automation also facilitates data collection and process

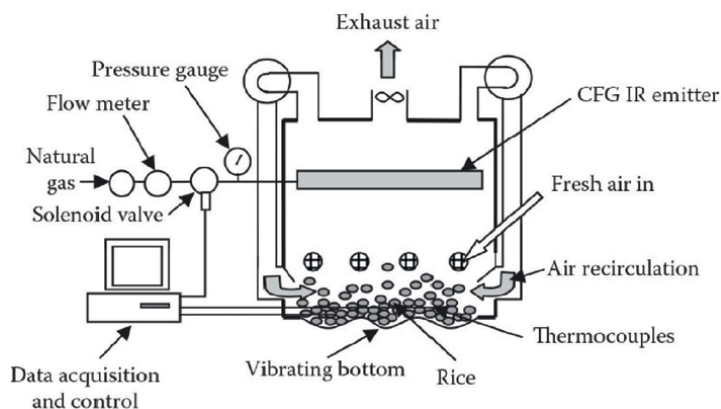


Figure 5.
Schematic of a simultaneous IR drying and disinfestation system.

optimization, ensuring regulatory compliance and consistent results across a diverse range of food products, from grains to vegetables and snacks. The *simulation* demonstrated the reliability of the fuzzy control model, achieving satisfactory computational efficiency [51]. The design of *decision-support systems* (DSS) based on fuzzy rules and data mining capable of improving the controllability of infrared drying processes, particularly in relation to maximizing the sensory quality of dried foods, represents one of the notable approaches for advancing the development of *intelligent drying equipment* [52].

4. Applications in food processing

4.1 Grains and cereals

Infrared drying is revolutionizing grain and cereal processing by providing precise control over drying parameters, which is essential for retaining their structural integrity and sensory qualities. Grains, such as wheat, rice, and barley, benefit significantly from uniform heat distribution, ensuring consistent moisture removal across kernels. This method prevents over-drying, which often leads to cracking and diminished texture in conventional methods [44, 53, 54]. Studies have highlighted the improved gelatinization properties in rice dried with infrared technology, enhancing its texture and cooking behavior. Over an 8-month storage period at 35°C, the water uptake ratio and volume expansion ratio of rice subjected to IR drying were 13.5 and 74.3% lower, respectively, compared to air-dried rice. Consequently, IR drying helps mitigate protein oxidation in stored rice, enhances water absorption capacity, promotes kernel expansion during gelatinization, and improves the cooking quality and texture of rice [55]. These enhancements cater to the growing demand for high-quality cereal products in both industrial and consumer markets [55–57]. Additionally, infrared drying improves microbial safety by eliminating hotspots that can harbor harmful contaminants. The absence of such inconsistencies ensures safer and longer-lasting grain products. This benefit is especially critical for grains stored in regions with high humidity, where fungal contamination poses a significant risk [54, 58].

4.2 Fruits and vegetables

Infrared drying has demonstrated significant advantages in processing fruits and vegetables, offering precise moisture control while preserving their natural attributes. The rapid heat transfer minimizes shrinkage and helps maintain color, texture, and flavor, which are critical for consumer appeal. The preservation of bioactive compounds, such as antioxidants and vitamins, is another advantage of infrared drying [59]. Simultaneous infrared dry-blanching and dehydration (SIRDBD) using the intermittent heating method reveals that enzyme inactivation increases with prolonged heating, while blanching time decreases as surface temperature rises. Enzyme inactivation is significantly faster at a surface temperature of 80°C. Higher temperatures or increased sample thickness substantially raise the kinetic constant (k) for vitamin degradation, with greater activation energy noted in thicker samples. Vitamin C levels demonstrate consistent trends across different temperatures during initial radiation. The high-temperature-short-time (HTST) method is optimal for efficient IR blanching. Following polyphenol oxidase inactivation, dehydration can continue at lower temperatures to preserve Vitamin C content and maintain the nutritional quality of fruit slices [51, 60]. Fractional conversion modeling of color changes revealed that the degradation of natural pigmentation in fruits is strongly influenced by the interaction between fruit thickness and energy input. For thinner slices, applying lower temperatures is critical to preserving color quality, underscoring the importance of precise temperature control during processing [49]. Rehydration capacity evaluates the ability of a dehydrated product to regain its original properties by interacting with a liquid phase. This complex process involves three simultaneous steps: water absorption, product swelling, and the diffusion of soluble components. Moisture reabsorption depends on the texture and structure of the dried product. Infrared drying under mild thermal conditions minimizes structural breakdown, particularly in thinner slices, by reducing shrinkage coefficients and preserving the product's internal physical integrity [61]. Textural profile analysis (TPA) indicated infrared drying of fruits and vegetables reveals key textural changes with moisture loss. Adhesiveness decreases due to reduced free water, while springiness declines at higher temperatures, likely from a glass transition altering elasticity. Cohesiveness initially rises during softening but diminishes during hardening, depending on temperature. Gumminess, linked to the energy for disintegration, varies with texture, indicating "firm" or "soft" characteristics. Chewiness increases as dried samples harden, requiring more energy for mastication, while resilience improves with moisture loss. Thicker slices reduce cohesiveness, springiness, and chewiness. Optimal texture and consumer acceptance occur under conditions of controlled, low water evaporation rates [62].

4.3 Meat and seafood

Dehydration methods play a crucial role in the efficiency of beef jerky production. Mid- and far-infrared (MFIR) drying has demonstrated significant advantages over hot air drying by enhancing dehydration efficiency while reducing both time and energy consumption. This method also facilitates myofibrillar protein denaturation, promotes the release of immobile water as free water from myofibrils, and lowers the activation energy required for dehydration. With its superior efficiency and potential for optimization, the MFIR drying method presents a promising future for

high-performance meat processing applications [63]. However, despite its ability to significantly reduce processing time, the accuracy of infrared drying for meat products may be lower, likely due to the formation of a surface layer that restricts moisture release [64]. Infrared drying has proven to be a highly efficient method for processing seafood, offering additional advantages beyond those observed in meat products. It maintains the nutritional and sensory qualities of seafood, significantly reduces drying time, improves rehydration capacity, and enhances the color and texture of products like squid filaments and sea cucumbers. Moreover, it consumes less energy and minimizes surface damage, making it particularly suitable for thin-layer drying of high-quality seafood [65].

4.4 Snack foods and ready-to-eat products

Infrared-based combined processes have emerged as a promising technology for producing healthy, high-quality snack foods, offering significant advantages in efficiency, product quality, and energy savings. Sequential infrared blanching combined with hot air drying has been shown to enhance the production of crispy carrot snacks by reducing drying time and retaining texture and nutrients [66]. Infrared-assisted freeze-drying (IRAFD) has demonstrated its potential for strawberry and banana snacks by achieving faster drying rates, preserving color, flavor, and bioactive compounds, and improving energy efficiency compared to traditional methods [67, 68]. Additionally, infrared drying combined with air frying has been effectively applied to tilapia skin-based ready-to-eat snacks, improving physicochemical and sensory properties, such as texture, flavor, and nutritional value [69]. It should be noted that for a better understanding of the principles of infrared drying, simulation studies of this process have been conducted on apple- and potato-based products as common snacks [51, 70]. Overall, the integration of infrared technology in snack food processing minimizes nutrient loss, ensures consistent product quality, and reduces energy consumption, making it an ideal choice for sustainable industrial applications [66–70].

5. Practical insights into infrared technology

5.1 Economic considerations

The cost of a dehydration process depends on several factors, including capital costs, labor costs, energy costs, and the energy efficiency of the system [71]. Infrared drying is widely recognized for its energy efficiency due to its ability to directly transfer heat to the product surface, minimizing energy losses. Comparative studies have consistently shown that infrared drying consumes significantly less energy than traditional hot air drying methods. The infrared dryer demonstrated an energy efficiency of 35–45% [37]. More than 85% of dryers used in the food industry are conventional hot air dryers, which account for up to 20% of the total energy consumption in food production. These dryers have an energy efficiency of approximately 30% and contribute to 90% of the overall production costs. Additionally, up to 45% of the energy input in these systems is lost as waste heat through exhaust gases, leading to reduced efficiency and increased greenhouse gas (GHG) emissions [72]. Moreover, the shorter drying cycles enabled by infrared systems enhance productivity while reducing operational costs. These advantages make it an appealing choice for

industries seeking efficient and sustainable drying solutions [72, 73]. Additionally, integrating infrared process with other costly drying methods, such as freeze-drying, can significantly reduce their overall costs [71]. The *techno-economic analysis* of infrared drying highlights its potential to revolutionize food industry drying processes by combining technical feasibility with significant economic benefits. Infrared drying demonstrated a high efficiency of 44% and the shortest drying time of 2.5 hours, resulting in reduced energy and labor costs. With a specific energy consumption (SEC) of just 1.37 kWh/kg of water evaporated, it is a highly energy-efficient option. Despite a higher initial investment, its payback period of 2.05 years and a benefit-cost ratio (BCR) of 1.08 confirm its economic viability. Furthermore, infrared drying enhances product quality by preserving moisture, texture, and microbial safety, making it a sustainable and cost-effective choice for commercial applications [74]. Similarly, this study was conducted for solar drying, focusing on evaluating its efficiency and economic viability [75]. From a market demand perspective, the applications of IR heating are expected to expand as consumers increasingly seek safe products with enhanced nutritional value and superior organoleptic quality. The comparative performance analysis of Far-infrared and conventional heating ovens shows that FIR ovens are significantly more efficient. They consume only 26.7% of the calories used by conventional ovens, maintain a 100% production rate, reduce processing time to 66.7%, and lower energy costs to 54.5% of conventional ovens. In all these aspects, Far-infrared technology demonstrates superior performance. Finally, despite its economic advantages, *insights for industrial implementation* emphasize that adopting infrared (IR) technology in the food and agricultural industries requires overcoming challenges, such as limited understanding and optimizing system designs to control surface temperature, prevent overheating, and ensure consistent product quality [76].

5.2 Environmental sustainability

The environmental impact of drying processes, including infrared (IR) drying, highlights its potential to mitigate greenhouse gas (GHG) emissions compared to conventional methods. Infrared drying significantly reduces energy consumption while maintaining product quality, thereby lowering carbon footprints in food processing. Studies provide practical insights into the importance of modern technologies, such as infrared and hybrid solar drying, in replacing traditional energy-intensive systems reliant on fossil fuels. These advancements support sustainability by minimizing carbon dioxide emissions, conserving natural resources, and aligning with global efforts to combat climate change and promote environmentally friendly food processing methods [77]. From an environmental sustainability perspective, IR drying offers notable advantages, including higher energy efficiency and reduced specific energy consumption (SEC). By decreasing reliance on fossil fuels, especially when paired with renewable energy sources, IR drying effectively lowers GHG emissions. Additionally, enhanced process control reduces heat losses, further contributing to environmental sustainability. These features establish IR drying as a cleaner, more eco-friendly option for industrial food processing [78].

5.3 Regulatory and safety standards

Infrared drying processes are governed by stringent food safety requirements to ensure the *food safety* and quality of dried products. The effectiveness of microbial

inactivation through infrared (IR) heating is influenced by several factors, including the infrared power level, heating rate, peak wavelength, and bandwidth of the IR source. Additionally, it depends on the physical shape and size of the product, the type and physiological phase of microorganisms (such as exponential growth or stationary phase), and the type of food material being treated [79]. The Code of Federal Regulations (CFR) serves as a compilation and codification of general, permanent, and customary regulations published in the Federal Register by federal government agencies, regulatory departments, and organizations, offering legal guidelines for the application of any novel methods in the food industry [80]. Researchers, in process design, utilize data mining techniques as part of Knowledge Discovery of Data (KDD) to analyze and select treatments within a structure that adheres to regulatory requirements [81, 82]. The electromagnetic spectrum differentiates between non-ionizing radiation (such as ultraviolet light, visible light, infrared radiation, and microwaves) and ionizing radiation (such as X-rays and gamma rays), each possessing unique properties, including wavelength, energy levels, penetration depth, and impacts on biological matter [83]. Under the regulation 29 *CFR 1910.1096*, the distinction between ionizing and non-ionizing radiation is explicitly stated, identifying infrared radiation as a category of non-ionizing radiation. Unlike ionizing radiation, infrared cannot ionize atoms but can increase the thermal energy of biological systems and materials [84]. According to the regulation 21 *CFR 890.5500*, the design of an infrared lamp focuses on emitting energy within the infrared frequency range (approximately 700–50,000 nanometers) to generate surface-level heat [85]. Despite the total safety of infrared devices in food drying, there are certain concerns that need to be addressed, which are highlighted here. Infrared radiation (IR) presents significant safety concerns, particularly in occupational and industrial environments, due to its thermal effects on biological tissues. Short-wavelength IR (<1500 nm) penetrates deeper, reaching the dermis and sometimes even deeper structures, while longer wavelengths are absorbed superficially, mainly in the epidermis. Acute effects on the skin include burns, increased vasodilation of capillary beds, and pigmentation changes that can persist over time. Continuous exposure to high-intensity IR radiation may lead to permanent erythema and chronic inflammation. Factors influencing the severity of these effects include exposure intensity, duration, and the ability of tissues to dissipate heat. For example, skin regions with high pigmentation or inadequate circulation are more prone to thermal damage. The eyes are particularly vulnerable to IR radiation. The cornea, transparent to IR wavelengths between 750 and 1300 nm, allows significant energy transmission to deeper structures like the lens and iris. Corneal damage occurs at doses as low as 7.6 W.s/cm² (watt-seconds per square centimeter) for wavelengths between 880 and 1100 nm, while the iris can sustain injuries at doses requiring corneal exposures around 10.8 W.s/cm². Damage includes protein coagulation, corneal opacification, and cataract formation. Studies have confirmed that IR-induced cataracts are primarily due to local heating effects in the lens caused by energy absorption from the iris and cornea. Regulations, such as the *Radiation Control for Health and Safety Act* (1968) and the *Occupational Safety and Health Act* (1970), enforce strict standards for managing IR exposure in occupational settings. These include emission limits, monitoring protocols, and mandatory safety measures for devices emitting IR radiation. Thresholds, such as corneal exposure limits of 7.6 W.s/cm² and occupational exposure guidelines for high-temperature environments, help prevent acute and long-term injuries. However, challenges remain in fully understanding the chronic effects of repeated IR exposure and individual variability in tissue responses. Ongoing research is necessary to refine safety standards and improve protective strategies to ensure worker and public safety [86–90].

6. Conclusions and outlook

Infrared drying represents a significant advancement in food processing technology, offering clear benefits over traditional methods. Its ability to enhance energy efficiency, reduce drying times, and retain the quality of food products makes it a versatile and valuable tool across a wide range of applications. From grains and fruits to vegetables and meats, this technology has proven to be adaptable to diverse food processing needs while ensuring superior outcomes. Practical insights into the implementation of infrared drying have highlighted its potential to achieve precise heat distribution and tailored drying conditions. These capabilities ensure minimal thermal degradation and consistent product quality, making it particularly advantageous for high-value and delicate food items. Such insights also underscore the benefits of integrating infrared drying into existing processes to improve efficiency and optimize operational costs. As infrared drying technology continues to evolve, integrating advanced systems such as automation and hybrid methods has expanded its scope and efficiency. By combining infrared drying with other techniques, such as vacuum or microwave drying, tailored solutions for various food matrices have become achievable. However, further research is essential to refine these combinations and address challenges related to scalability, energy optimization, and the preservation of bioactive compounds. Looking forward, the focus on sustainability presents opportunities for integrating renewable energy sources into infrared drying systems.

Conflict of interest

The authors declare no conflict of interest.

Notes

This chapter is crafted for researchers and practitioners who seek to transform theoretical knowledge into impactful industrial applications. It highlights the importance of bridging science and practice, emphasizing that knowledge achieves its true potential only when effectively implemented. By providing practical insights into infrared drying, this chapter equips professionals with the tools to address real-world challenges in food processing. It serves as a guide for turning innovative concepts into actionable solutions, ensuring progress and excellence in industrial operations.

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Effects of Low-Frequency Ultrasound on the Drying Process, Microstructure, Surface Chemistry, and Crystallinity of Milk Powders with Varying Casein-to-Whey Ratios and Calcium Addition

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Abstract

This study investigates the effects of casein-to-whey protein ratios, ultrasound pre-treatment, and calcium addition on the surface composition, microstructure, and crystallinity of spray-dried milk powders. X-ray photoelectron spectroscopy (XPS) showed that protein is the predominant component on the powder surface, with sonication enhancing protein surface activity while reducing lactose segregation. Calcium addition increased the presence of oxygen-containing functional groups and stabilised the surface structure. Sonication reduced particle size and smoothed surface morphology, as observed by scanning electron microscopy (SEM), particularly in whey protein samples, whereas casein-rich formulations showed rougher, more irregular structures, making them more resistant to ultrasound-induced fragmentation. X-ray diffraction (XRD) analysis indicated the lactose predominantly remained in an amorphous state across all samples. Calcium addition further promoted the amorphous structure by forming complexes with proteins and lactose. Sonication had a minimal impact on lactose crystallinity, but it played an important role in enhancing protein surface activity and modifying surface composition.

Keywords: ultrasound, milk proteins, lactose, calcium, spray-drying, powder

1. Introduction

Dairy powders are foundational ingredients in the food industry, known for their stability, convenience in transportation, and wide-ranging applications, from infant formulas to functional foods and nutraceuticals [1, 2]. The transformation of liquid milk into powder through drying process not only extends shelf life but also facilitates

the integration of milk's nutritional and functional benefits into diverse product formulations. For dairy powders to deliver their intended functional characteristics, they must first undergo rapid and thorough rehydration, making complete reconstitution a necessary step prior to use [3].

Spray drying is the widely used dehydration method that rapidly removes water from atomised droplets exposed to hot air [2]. The rapid drying process influences the microstructure and surface composition of milk powders by redistributing components such as proteins, lactose, fats, and minerals [1, 4, 5]. Surface formation in milk powder particles is generally attributed to two mechanisms: surface-active substances interacting at the air/liquid interface [6] and solid-solute segregation [7]. Under both mechanisms, proteins and lipids preferentially accumulate near the surface, especially in the first 5 nm of the particle [7–9].

Lactose, the predominant carbohydrate in milk, plays a significant role in the quality and stability of dairy powders [10]. Its physical state, whether amorphous or crystalline, critically influences powder behaviour, including solubility, moisture uptake, and caking. Crystallised lactose, particularly in the form of α -lactose monohydrate, contributes to the stability of the powder, while amorphous lactose, though more soluble, is highly prone to moisture absorption. This absorbed moisture increases molecular mobility to the point where amorphous lactose can crystallise into α -lactose monohydrate under specific conditions, such as high humidity or storage over time [11]. The instability of amorphous lactose in dairy powders is closely linked to storage and handling issues, including stickiness and caking [12–14]. The crystallisation of lactose during drying and/or storage is a complex process, influenced by interactions with proteins and minerals, which can act as a nucleating agent or inhibit crystallisation depending on its concentration and the processing conditions [15–18]. Studies have shown that the presence of whey proteins promotes nucleation but slows down the growth of lactose crystals [19, 20]. Similarly, whey protein isolate has been reported to act as a stabiliser in sugar-protein systems during spray drying and freeze-drying [21]. Additionally, the effect of calcium on the rate of lactose crystallisation or the growth rate of lactose crystals has been studied across various systems [22, 23]. Moreover, the addition of calcium and its interactions with milk proteins further complicate the microstructure and crystallinity of the powder. The presence of calcium-protein interactions further complicates lactose crystallisation and alters the structural integrity of the powder, impacting solubility and rehydration behaviour [24].

Ultrasound processing is an emerging non-thermal technology that modifies the structural and functional properties of food materials [25, 26]. In dairy systems, ultrasound has been shown to influence the aggregation of proteins, enhance solubility, and modify the crystallisation behaviour of lactose, potentially improving the functional properties of the resulting powders [27, 28]. Our recent study on protein-lactose-calcium powders showed that short-term sonication improved solubility through enhanced particle dispersion, whereas prolonged sonication led to fragmentation and reduced solubility [29]. Calcium stabilised particle size in casein-rich systems but promoted aggregation in whey-dominant formulations. While previous studies have explored the effects of spray drying on milk components and the impact of ultrasound on lactose-protein, lactose-mineral, protein-mineral interactions, there is a lack of detailed understanding of how ultrasound treatment prior to drying influences the microstructure, surface chemistry, and lactose crystallinity of protein-lactose-calcium systems with varying casein-to-whey protein ratios. The aim of the present study is to investigate these effects using low-frequency ultrasound, applied

before spray drying, on protein-lactose powder systems both with and without the addition of calcium. The research could lead to better control over lactose crystallisation, optimised protein ratios in dairy formulations, and new processing techniques that improve the quality and functionality of dairy powders.

2. Materials and methods

2.1 Materials

Bovine Milk Protein Concentrate (MPC; ~85.5% protein: 67.5% casein, 16.2% whey; 1.5% fat, 8.0% ash, $\leq 5.5\%$ moisture) and Whey Protein Concentrate (WPC; 1.0% fat, 2.5% ash, $\leq 4.7\%$ moisture) were supplied by Fonterra Co-operative Co. Ltd. (Richmond, VIC, Australia). Calcium chloride dihydrate (molecular weight: 147.01) and α -Lactose monohydrate powder (99.6% purity) were purchased from Sigma-Aldrich Pty. Ltd. (Castle Hill, NSW, Australia). All samples were prepared using Milli-Q ultrapure water as the solvent.

2.2 Preparation of concentrated samples

Concentrated solutions (20% total solids, w/w) were prepared with three casein (CN) to whey protein (WP) ratios: 60:40 (64 M), 50:50 (55 M), and 0:100 (WP), following our previously described method [30]. The total protein content was kept constant across all formulations (6.6% w/w, dry basis), with the CN:WP ratio adjusted by blending the respective protein powders. Lactose was added to all samples at 5% (w/w, included in the 20% total solids). Calcium chloride was incorporated only into designated formulations, at a final concentration of 30 mM, and was not included in the calculation of the 20% total solids. The mixtures were stirred magnetically at room temperature for 30 min to achieve uniform dispersion, and then stored at 4°C for 12 h to allow complete hydration. After hydration, the samples were subjected to ultrasound treatment and subsequently spray-dried under identical operating conditions, as described in the following sections.

2.3 Low-frequency ultrasound treatment

Ultrasound pre-treatment was conducted on 500 mL reconstituted concentrates using a low-frequency ultrasound (20 kHz) unit with a 12-mm probe at 40% amplitude for 0, 1, and 5 min, corresponding to energy density of 5.22 and 26.08 J/mL respectively. An ice bath was used to hold sample temperatures under 10°C, and readings were recorded after each treatment with a digital thermometer (Brannan, Australian Scientific Pty Ltd., NSW, Australia).

2.4 Spray-drying

Sonicated and non-sonicated concentrates were spray-dried using a laboratory-scale spray dryer B290 (Buchi Labortechnik AG, Flawil, Switzerland), under conditions: inlet 180°C, outlet 80°C, feed flow 16 mL/min, nozzle diameter 0.7 mm, and airflow 35 m³/h. Prior to drying, all samples were equilibrated in a water bath to $20 \pm 1^\circ\text{C}$ to standardise feed temperature. Powders were sealed in airtight containers, placed in a desiccator at room temperature, and analysed within 2 h to limit moisture absorption.

2.5 XPS analyses

2.5.1 Equipment

Surface composition of the powders was characterised using a Thermo K-Alpha X-ray Photoelectron Spectrometer (Thermo Fisher Scientific, East Grinstead, UK). Samples were prepared by attaching the powdered material onto silicon pieces with double-sided carbon tape. The mounted samples were stored overnight in a vacuum desiccator before measurement. A wide-scan spectrum was first obtained with a pass energy of 160 eV, a step size of 1 eV, and a dwell time of 50 ms. High-resolution spectra were then recorded with a reduced pass energy of 20 and 0.1 eV step size while maintaining the same dwell time [31]. For data interpretation, it was assumed that milk powders mainly consist of carbohydrates, proteins, and fats. The atomic percentages of carbon, nitrogen, and oxygen in the outer layer (approx. 10 nm) of the particles were measured, and these values were used in a matrix calculation to estimate the relative contributions of the three main components.

2.5.2 Use of XPS for surface composition of lactose, protein, and fat

Spectral analysis was carried out using CasaXPS software. Background subtraction was performed with a Shirley baseline, and the peaks were fitted with a mixture of 70% Gaussian and 30% Lorentzian functions [1]. Quantification relied on photoemission cross-sections and transmission factors provided within the software. The detailed methodology has been described previously [6]. In brief, the atomic concentrations of carbon, oxygen, and nitrogen were determined and incorporated into a matrix model to calculate surface levels of lactose, protein, and fat:

$$IC = \alpha PICp + \alpha' LICLa + \alpha'' FICLi \quad (1)$$

$$IO = \alpha PIOp + \alpha' LIOLa + \alpha'' FIOLi \quad (2)$$

$$IN = \alpha PINp + \alpha' LINLa + \alpha'' FINLi \quad (3)$$

The I^C , I^O , and I^N represent the mole fractions of carbon, oxygen, and nitrogen at the surface, obtained from the peak areas of C1s, O1s and N1s [1]. The surface proportions of protein, lactose, and fat were then determined by solving the equations for αP , $\alpha' L$, and $\alpha'' F$.

2.6 Scanning electron microscopy (SEM)

Powder morphology was studied with a field emission scanning electron microscope (SEM; FEI Quanta 200, Tokyo, Japan) fitted with a tungsten filament. About 0.1–0.2 g of sample was mounted on stainless steel stubs and iridium-coated with a Leica EM ACE600 sputter coater (Leica Microsystems, Wetzlar, Germany). Imaging was conducted at 30 kV accelerating voltage, with magnifications of 500 $\times$, 1000 $\times$, and 3000 $\times$ to visualise surface structural features in detail.

2.7 X-ray diffraction analysis (XRD)

The crystalline nature of lactose was evaluated using a Bruker Axs D4 Endeavor wide-angle X-ray diffractometer (Bruker, Bremen, Germany) operated at 35 mA and

40 kV. Approximately 1.5 g of powder was spread onto a stainless-steel holder and exposed to Co-K α radiation. Diffraction data were collected over a 10–30° 2 θ range, with 0.02° increments, and a total scan time of 19 min [32]. Diffrac.Eva.V4.3 software (Bruker) was used to identify lactose peaks using a reference database. Degrees of crystallinity were determined from the characteristic peaks, including 20.0° (α -lactose monohydrate), 21.0 and 10.5° (anhydrous β -lactose), and 19.2°/19.7° (α - and β -lactose at molar ratios of 5:3 and 4:1) [33–35].

2.8 Statistical analysis

All experiments were repeated three times, with at least duplicate measurements for each replicate. Data were analysed using one-way ANOVA, followed by Tukey's post hoc test to assess pairwise differences. Differences were considered significant at $p < 0.05$. All statistical analyses were conducted using OriginPro 2021b (OriginLab Corporation, Northampton, MA, USA).

3. Result and discussion

3.1 Surface chemical composition

The surface chemical composition of spray-dried milk powders was analysed using XPS to quantify the contributions of protein, lactose, and fat. The binding energies of C1s (281–293 eV), N1s (397–408 eV), and O1s (528–533 eV) align with the previously reported values [1]. The relative amounts of these components were calculated from their elemental percentages (Appendix A). The behaviour of spray-dried powders during handling, storage, and use is largely controlled by their surface composition, which affects traits like flowability, cohesion, stickiness, hygroscopicity, caking, wetting, rehydration, and dispersion [3, 4, 11].

The ternary plots (**Figure 1a–c**) illustrate the surface composition of spray-dried milk powders, highlighting the effects of sonication time and calcium addition on WP, 55 M, and 64 M formulations. Across all samples, protein remains the primary surface component, while lactose and fat distribution vary with formulation and treatment conditions. Without sonication, surface composition reflects casein-to-whey protein ratio differences. The WP samples show the highest protein coverage (75.48%)

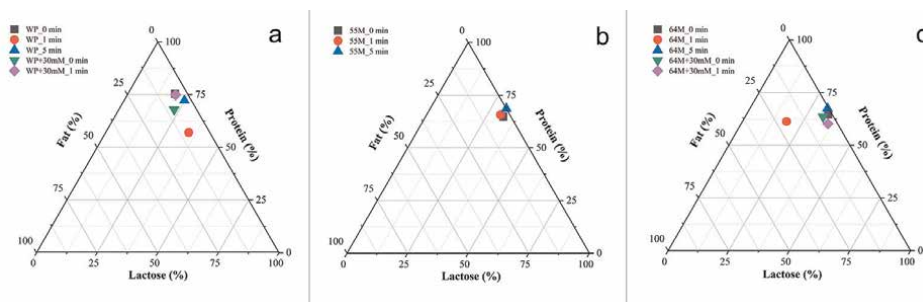


Figure 1. Surface composition of spray-dried milk powders with varying casein-to-whey protein ratios (WP: 0:100, 55 M: 50:50, 64 M: 60:40), produced with and without calcium addition (30 mM) and subjected to different ultrasound times (0, 1 and 5 min), as determined by XPS. Symbols in each ternary plot represent the combined influence of ultrasound and calcium treatments for the corresponding protein ratio.

compared with 55 and 64 M samples. Lactose coverage (19.35%) is the lowest in WP samples, while fat remains minimal. In contrast, 55 M samples present a more balanced profile, with lower protein coverage than WP but higher lactose content (32.07%), indicating that equal proportions of casein and whey promote surface coexistence of these components, with a limited fat presence. The 64 M samples shift toward lactose dominance (33.72%), with protein coverage (64.67%) similar to 55 M, but the highest lactose-to-protein ratio, suggesting that casein-lactose interactions drive migration to the surface.

The air-water interface during spray drying plays a key role in determining the surface composition of the final powder. According to Faldt and Bergenstahl 1994 [6], the most surface-active components concentrate at the droplet interface, predominating on the dried powder surface. Water diffuses out the droplets and evaporates, leading to the migration of various components within the droplet. Fat and protein, due to their larger size, hydrophobic nature, and slower diffusion rates, tend to concentrate at the surface, whereas lactose and minerals, which are smaller and more hydrophilic, migrate toward the droplet interior [7]. Given that casein is recognised as more surface active than whey proteins [36, 37], its presence at the interface likely influences lactose distribution. Unlike whey proteins, which form compact globular structures, casein micelles create a more open and flexible network, facilitating lactose migration. Several additional potential mechanisms may contribute to the enhanced lactose presence at the surface in high-casein formulations in this study. Firstly, casein micelles exhibit strong water-binding capacity, forming a hydration shell that slows water evaporation during spray drying. This prolonged moisture retention may allow lactose to remain dissolved in the mobile phase longer, increasing its probability of migrating toward the surface. This phenomenon aligns with our previous studies showing that an increase in casein ratio correlates with a decrease in T_2 relaxation time, indicating reduced molecular mobility and greater water retention [30, 38]. Secondly, casein's amphiphilic nature enables it to interact with both hydrophilic and hydrophobic molecules [39], potentially acting as a carrier for lactose molecules to reach the interface more efficiently. Another possible explanation involves electrostatic interactions; casein has a net negative charge at neutral pH, which may influence lactose movement through weak charge interactions, leading to preferential accumulation at the surface.

Sonication time significantly alters surface composition across all ratios. Protein coverage increases with sonication time in all samples. WP protein rises from 57.04 to 72.22%, 55 M from 64.74 to 68.57%, and 64 M from 61.33 to 67.41%. Short sonication shows slight reductions in protein due to initial aggregate disruption, but prolonged treatment restores and improves protein surface coverage. Lactose content varies with sonication time depending on the ratio. In WP samples, short sonication increases lactose to 34.28%, possibly due to the redistribution of smaller lactose molecules to the surface. However, with prolonged sonication, lactose decreases to 24.88%. In contrast, for 55 and 64 M samples, lactose initially drops slightly with short sonication but stabilises at 31.43 and 32.04%, respectively, after prolonged treatment.

Calcium addition generally increases surface lactose and fat while reducing protein coverage. In WP + 30mM_0, protein decreases to 67.04%, with lactose rising to 27.16% and fat to 9.27%. Sonication enhances protein migration to the surface in calcium-enriched samples, as seen in WP 30 + 30mM_1, where protein increases to 74.89%, while lactose and fat decreased to 19.95 and 5.16%, respectively. This suggests that ultrasound facilitates protein migration and suppresses the surface segregation of lactose and fat, likely by disrupting protein aggregates and enhancing protein-calcium

interactions. A similar trend is observed in calcium-enriched 64 M sample. In 64 M + 30mM_0, protein content slightly decreased to 63.56%, while lactose and fat contributions increased to 35.81 and 4.35%, respectively, compared to 64 M0. These changes highlight calcium's role in enhancing lactose and fat segregation to the surface. With sonication (64 M + 30mM_1), protein content increased to 60.22%, while lactose and fat decreased slightly to 32.09 and 3.97%, respectively. The improved protein migration with sonication reinforces the hypothesis that ultrasound enhances protein surface activity by reducing aggregation and strengthening protein-calcium interactions, while simultaneously suppressing lactose segregation. These findings highlight that calcium addition generally promotes lactose and fat migration to the surface while reducing protein coverage. However, sonication counteracts these effects by improving the protein surface activity, reducing lactose and fat segregation, and enhancing protein-calcium interactions.

In this study, C1s spectral deconvolution demonstrated four main sub-peaks (C—C, C—COOH, C—O, C=O, and O—C=O) in all samples, corresponding to the functional groups of surface macromolecules, namely proteins, fats, and lactose. **Figure 2** shows the percentage distribution of surface functional groups (C—C/C—H, C—O, C=O, and O—C=O) in spray-dried powders, highlighting the influence of protein ratios, sonication, and calcium addition. The XPS analysis showed that surface functional groups in spray-dried powders are significantly influenced by the casein-to-whey protein ratio, sonication, and calcium addition (Appendix B Figure B1 (a,b)). Across all samples, C—C/C—H bonds were the dominant functional groups, accounting for 55–74% of the surface composition, with C—O bonds (19–28%) and C=O groups (7–17%) as secondary contributors, while O—C=O groups remained minimal (1–2%). Without sonication or calcium addition, the surface composition varied with the protein ratio. WP_0 showed the highest C—C content (65%) and the lowest C—O (20%), reflecting the hydrophobic nature of whey proteins. In contrast, 55M_0 and 64M_0 displayed slightly higher oxygen-functional groups, with C—O and C=O increasing to 22 and 7%, respectively, indicating casein's role in promoting surface oxidation. Sonication further modified the surface composition, particularly in WP samples, where increased sonication intensity reduced C—C content (from 65% in WP_0 to 55% in WP_5) and enhanced C—O (from 20 to 27%), suggesting that ultrasound processing promotes surface oxidation. The 55 M samples were less affected by sonication, with C—C and C—O contents remaining stable (70–71% and 22–23%, respectively), likely due to the balanced casein-to-whey ratio stabilising the surface chemistry. In 64 M samples, sonication effects were more variable, with C—C increasing to 74% in 64M_5 but decreasing to 57% in 64M_1, indicating complex interactions between casein and whey proteins under varying sonication conditions. Calcium addition promoted surface oxidation in both WP and 64 M samples, increasing C—O and C=O groups while reducing C—C content, as observed in WP + Ca_0 (C—C reduced from 65 to 63%) and 64 M + Ca_0 (C—C reduced from 72 to 70%). However, when calcium addition was combined with sonication, the oxidation effects were mitigated, as seen in WP + Ca_1 and 64 M + Ca_1, where C—C content slightly increased, and C—O and C=O decreased.

3.2 Surface microstructure and roughness

The surface morphology of milk powders was analysed using SEM (**Figures 3 and 4**). The microstructure of milk powders as observed through SEM varied significantly across formulations, particularly due to differences in casein-to-whey ratios

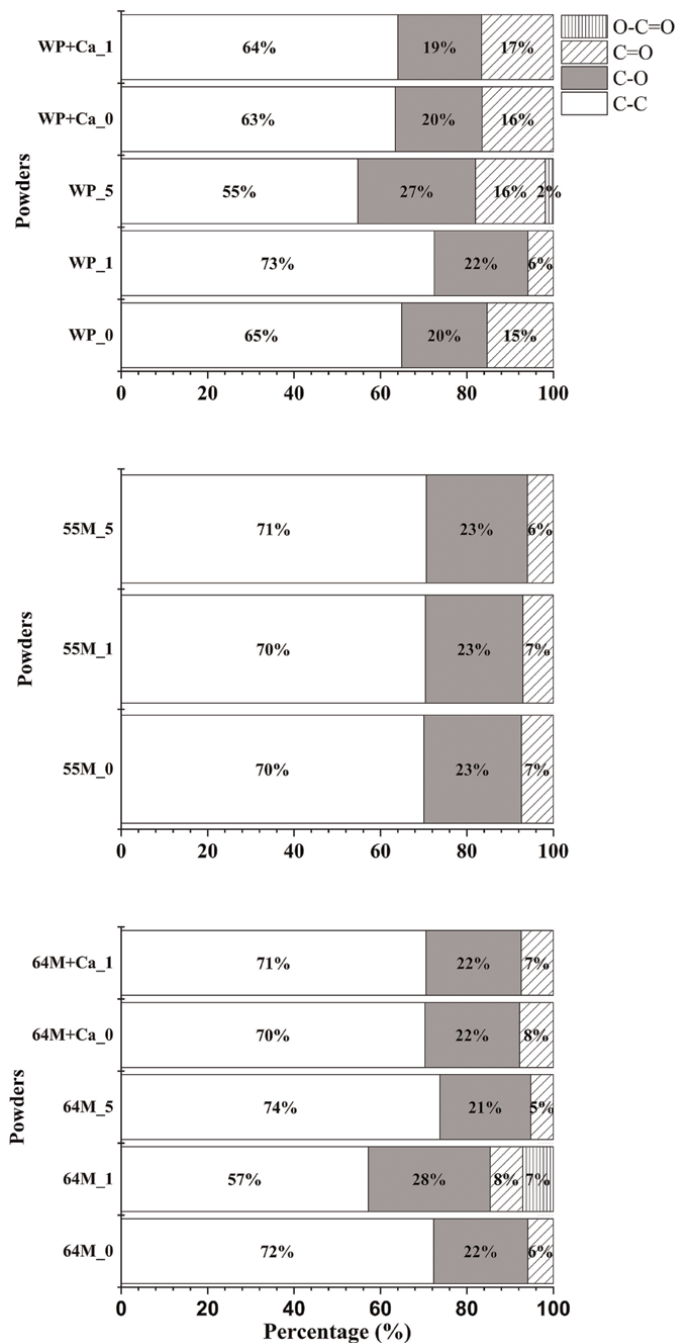


Figure 2. Distribution of surface functional groups (C—C/C—H, C—O, C=O, and O—C=O) on spray-dried powders analysed using XPS.

(**Figure 3**). The whey protein-only sample (WP) exhibited more defined and spherical particles, characterised by a smooth surface, compared to the 55 and 64 M samples. The WP formulation had fewer aggregated structures and a more uniform particle

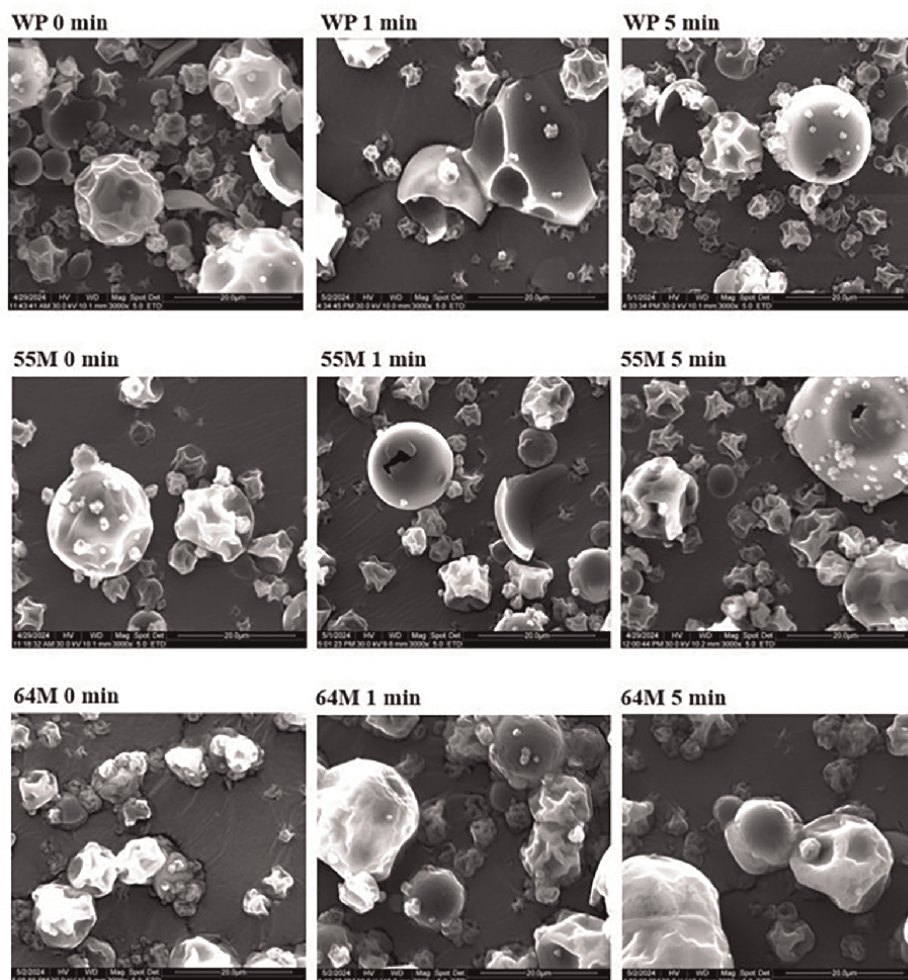


Figure 3.
 SEM images of milk powder particles under varying formulations and sonication times.

distribution. This could be attributed to the presence of whey protein, which tends to produce more discrete particles during drying [40, 41]. In contrast, the 55 M formulation showed a mix of both larger and smaller aggregated structures, reflecting the influence of both casein and whey components. The presence of casein, known for its tendency to form micellar structures, resulted in a more clustered arrangement compared to WP [42]. Meanwhile, the 64 M formulation, being more casein-rich, displayed larger and more irregular particles with a rougher surface. This observation aligns with the fact that casein's micellar structure creates a denser, less uniform particle shape, which contributes to the observed heterogeneity [42]. Similarly, the study by Mimouni et al. [42] observed that the casein micelles appeared to be linked either by direct intermicellar contacts or by short bridges composed of material protruding from the surface of micelles in milk protein concentrate powder samples. These SEM observations align with the XPS findings, where casein-rich samples (55 and 64 M) exhibited higher surface lactose content and oxygen-containing functional groups, indicative of casein's influence on surface chemistry and structure. The

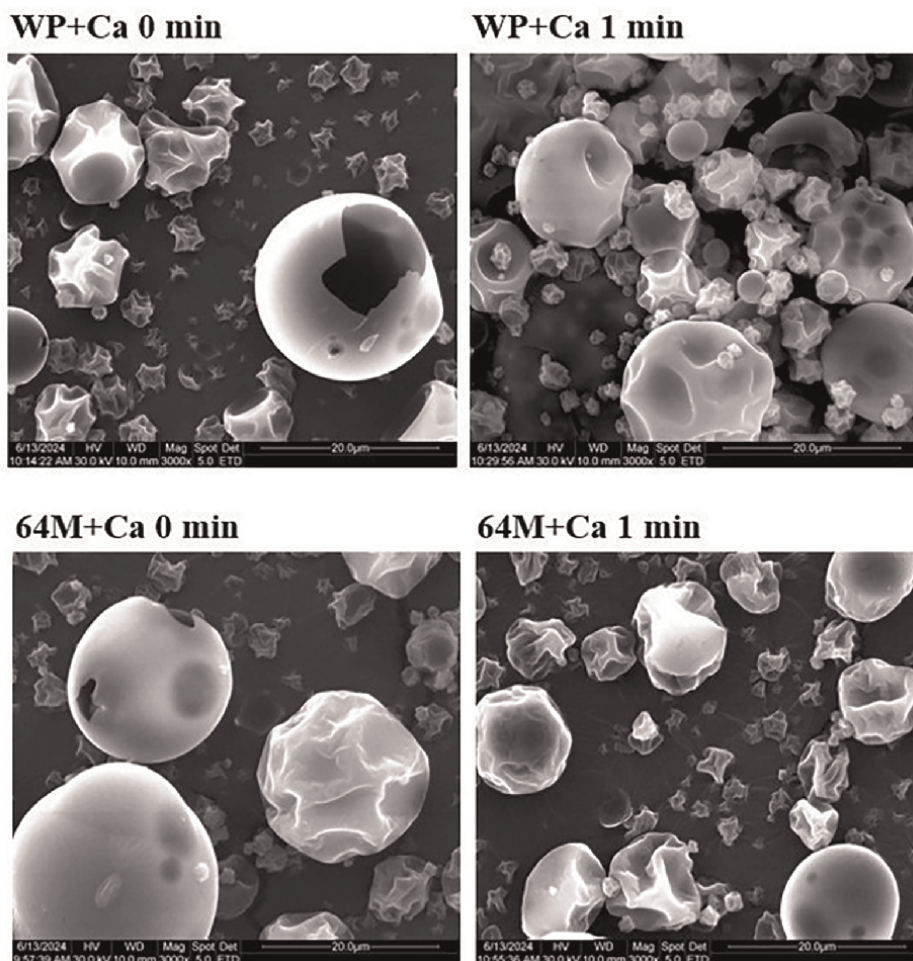


Figure 4. SEM images of particles with different morphology as a result of calcium addition, different formulations, and sonication times.

rougher and more irregular surfaces in casein-rich samples likely contributed to the observed surface oxidation and lactose enrichment during spray drying. Conversely, the smoother surfaces of WP samples correspond to the dominance of hydrophobic C—C/C—H functional groups and lower oxygen-functional groups, as revealed by XPS.

In addition, the influence of sonication treatment was evident in the microstructure of all the formulations, with different effects observed at different sonication durations (0, 1, and 5 minutes). In WP samples, sonication led to a marked reduction in particle size and increased surface smoothness, especially after 5 minutes of sonication. This suggests that ultrasound-induced cavitation disrupted the larger aggregates and smoothed out particle surfaces, resulting in finer and more homogenous structures [43]. For the 55 M formulation, sonication similarly resulted in fragmentation of the larger aggregated structures. After 1 minute of sonication, the particle surface began to show disruption, with smaller, more defined particles forming around the larger ones. By 5 minutes, there was an increase in smaller fragments, and

the surfaces of particles appeared rougher, indicative of more effective breakdown and dispersion of components with prolonged sonication. In the 64 M formulation, the effect of sonication was more subtle compared to WP. After 1 minute of sonication, there was increased surface irregularity, and the previously stable aggregates started fragmenting, leading to a more porous appearance. However, after 5 minutes, the 64 M samples still retained some larger fragments, which suggested that the casein network, due to its stable micellar structure, was more resistant to disruption by ultrasound compared to whey protein. This stability could be attributed to the denser, more interconnected micellar structure of casein, which requires higher energy for effective fragmentation. These morphological changes align with the XPS findings, where prolonged sonication in WP and 55 M samples enhanced protein surface activity, as evidenced by increased surface protein content and reduced lactose segregation. The smoother and finer WP particles observed after 5 minutes of sonication correspond to the dominance of hydrophobic C—C/C—H groups revealed by XPS, while the rougher and more fragmented 55 and 64 M surfaces reflect the influence of casein's micellar network on surface composition. The resistance of 64 M samples to ultrasound-induced fragmentation is consistent with XPS results showing the persistence of higher lactose and oxygen-functional group contributions, likely due to the casein's strong interactions and structural stability.

The addition of calcium had a significant impact on the microstructure of the milk powder formulations (**Figure 4**). Calcium acts as a cross-linking agent, which promotes structural cohesion. This effect was most noticeable in the WP formulation, where calcium addition resulted in more tightly bound aggregates, even at minimal sonication durations. SEM images of WP with 30 mM calcium at 0 minutes showed larger, more compact particles compared to the calcium-free sample. This suggests that calcium helped maintain particle cohesion, possibly through interactions between calcium ions and the protein matrix. In 64 M formulation, the addition of calcium contributed to the formation of more stable, cohesive particles, with fewer small fragments observed post-sonication. The presence of calcium seemed to reinforce the micellar structure of casein, making it more resistant to fragmentation by ultrasound. Even after 1 minute of sonication, the 64 M samples with calcium maintained a more compact and less disrupted structure, indicating that calcium effectively stabilised the protein network under mechanical stress. These observations also align with the XPS results, where calcium addition increased oxygen-functional groups (e.g. C—O and C=O), indicative of enhanced protein-calcium interactions. The structural cohesion observed in SEM is consistent with the increased surface hydrophilicity reported in XPS analysis, as calcium appears to promote the exposure of oxygen-containing functional groups while stabilising the protein network. The resistance of calcium-treated 64 M samples to fragmentation mirrors the persistence of higher lactose and protein contributions to the surface, further highlighting the stabilising role of calcium in both microstructure and surface chemistry.

3.3 Crystallinity

The XRD patterns of the spray-dried formulations show a largely consistent trend across all samples, regardless of casein-to-whey ratio, sonication time, or calcium addition (**Figure 5**, A–D). The broad diffraction peaks observed in all samples indicate that lactose predominantly remains in an amorphous phase, as evidenced by a halo pattern and the absence of distinct diffraction reflections. This observation aligns well with the previously reported patterns for amorphous lactose [44–49]. This result was

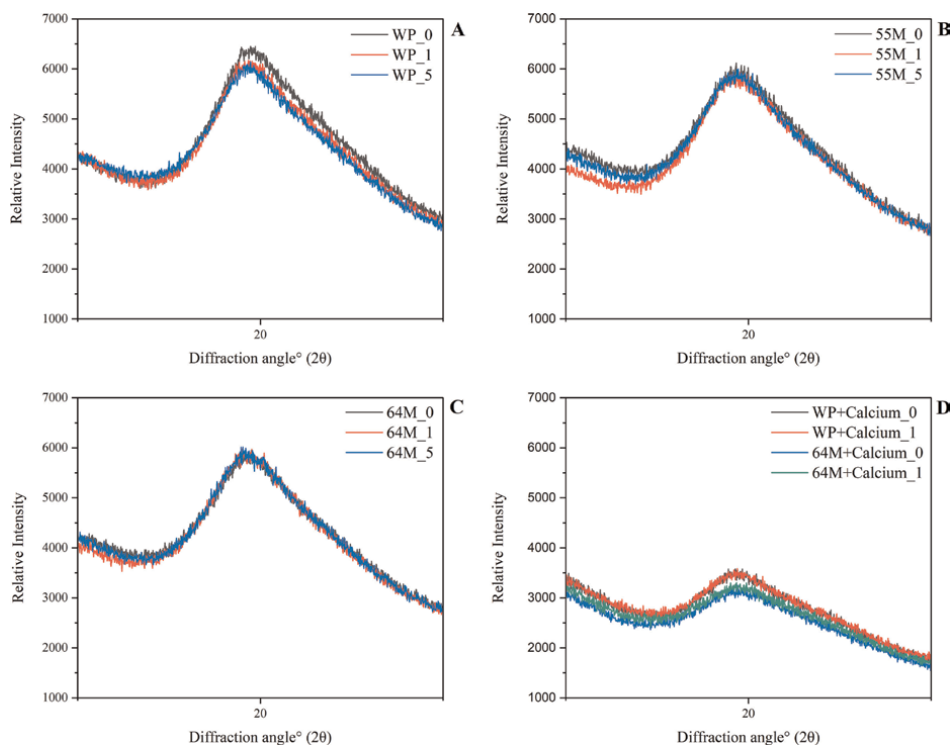


Figure 5.
X-Ray diffraction patterns for fresh spray-dried powder formulations.

expected, as lactose in spray-dried powders is generally amorphous [49, 50]. Spray drying lactose below its saturation point causes a rapid increase in viscosity, which inhibits crystallisation and promotes the production of amorphous lactose [51]. At drying temperatures above 50°C, mutarotation of lactose can occur, altering the α - to β -lactose ratio as concentration and temperature shift. Under these conditions, when the lactose solution becomes saturated, minor crystalline fractions may develop in samples that are predominantly amorphous [34]. This partial crystallisation is consistent with the observed XRD patterns, where an overall amorphous structure is maintained, but minor crystalline regions may still be present. These XRD findings complement the XPS results, where the surface composition of the powders reflected lactose contributions, particularly in casein-rich formulations (e.g., 55 and 64 M). The amorphous nature of lactose aligns with the SEM observations of heterogeneous surface morphology in these formulations, where surface irregularities may be linked to localised regions of amorphous lactose. Additionally, the stabilising effects of calcium and the structural changes induced by sonication observed in SEM may influence lactose crystallisation kinetics, further supporting the predominance of amorphous phases in these spray-dried powders.

Ultrasound treatment plays a role in influencing lactose structure in liquid phase. The cavitation forces generated during ultrasound may enhance molecular mobility and disrupt pre-existing crystals or disrupt agglomerates or nuclei [28, 52, 53], thereby promoting amorphous regions before the spray-drying step. In this study, although ultrasound did lead to slight variations in peak intensity, the general amorphous structure of lactose remained predominant across all sonication treatments. Notably,

the WP sample showed a higher peak intensity without sonication compared to the other samples. This might be attributed to the inherent characteristics of whey protein, which has been reported to promote lactose crystallisation to some extent [28]. It has been reported that hydrogen bonding occurs between polar groups of proteins and lactose under dry conditions, potentially creating competition with lactose-lactose interactions and affecting the crystallisation of lactose.

With calcium addition, the overall XRD patterns remained largely amorphous, but peak intensities decreased for both WP and 64 M samples, suggesting an increased tendency toward maintaining an amorphous state. The presence of calcium likely influences the lactose crystallinity by interacting with proteins and lactose molecules, forming complexes that prevent the orderly alignment required for crystal formation. This interaction between calcium and casein seems to enhance the amorphous character of the matrix, which aligns with findings that calcium can stabilise amorphous structures by creating a more cohesive network. This observation aligns well with study by Morgan et al. [54] that interactions between lactose and minerals might also impact the crystallisation of lactose. Ionic cations and anions can delay the lactose crystallisation through altering the structure of water [54]. Low-charge-density ions like Cl^- disrupt and destabilise the water network, whereas high-charge-density ions such as Ca^{2+} can reorganise the structure of water in lactose solutions [55]. The addition of CaCl_2 alters the solubility of lactose and modifies the crystal growth stage according to the concentration of the salt. Calcium ions, enclosed by a strong electric double layer, engage with four to six surrounding layers of water molecules *via* dipole-ion interactions, thereby limiting water mobility. This restricted mobility of water creates the necessity of high energy requirements for lactose crystallisation leading to low crystallisation rates [23]. Lactose hydroxyl groups can attach to the calcium hydration layer, producing calcium-lactose complexes. Such binding interferes with the ordered molecular arrangement required for crystallisation [23].

Overall, the findings indicate that while both ultrasound pre-treatment and calcium addition have significant effects on lactose crystallinity, the primary factor determining the amorphous nature of lactose is the rapid dehydration during spray drying. Calcium appears to further promote an amorphous state, and sonication introduces mechanical energy that could either disrupt or prevent crystal formation, though the latter effect remains subtle compared to the influence of spray drying. Sonication, on the other hand, introduces mechanical energy that can disrupt pre-existing crystals or prevent new crystal formation. However, the impact of sonication on lactose structure remains secondary to the dominant effect of rapid water removal during spray drying. Together, these processes highlight the interplay between formulation and processing conditions in shaping the structural characteristics of lactose in spray-dried powders.

4. Conclusion

This study highlights the significant influence of formulation and processing conditions on the surface chemistry, microstructure, and crystallinity of spray-dried milk powders. XPS analysis demonstrated that protein and lactose dominate the surface composition, while fat contributions remain minimal. Sonication enhanced protein dispersion and surface activity, reducing lactose segregation, with the effects being most pronounced in whey protein (WP) samples. In casein-rich formulations, sonication fragmented aggregates but retained some structural stability due to casein's

micellar network. Calcium addition further modified the surface composition by promoting the exposure of oxygen-functional groups and stabilising amorphous structures through protein-lactose interactions. SEM findings revealed that sonication reduced particle size and smoothed surfaces in WP, while casein-rich powders exhibited rougher and more irregular morphologies. XRD confirmed the amorphous nature of lactose, with calcium addition enhancing this state by restricting crystallisation through its interactions with lactose and water.

From a practical perspective, these findings suggest that ultrasound pre-treatment and targeted calcium addition can be used to control surface composition, particle morphology, and lactose crystallinity, thereby improving rehydration behaviour, structural stability, and shelf life of high-protein dairy powders. Such strategies are particularly relevant for applications in infant nutrition, sports formulations, and functional foods where powder stability and solubility are critical.

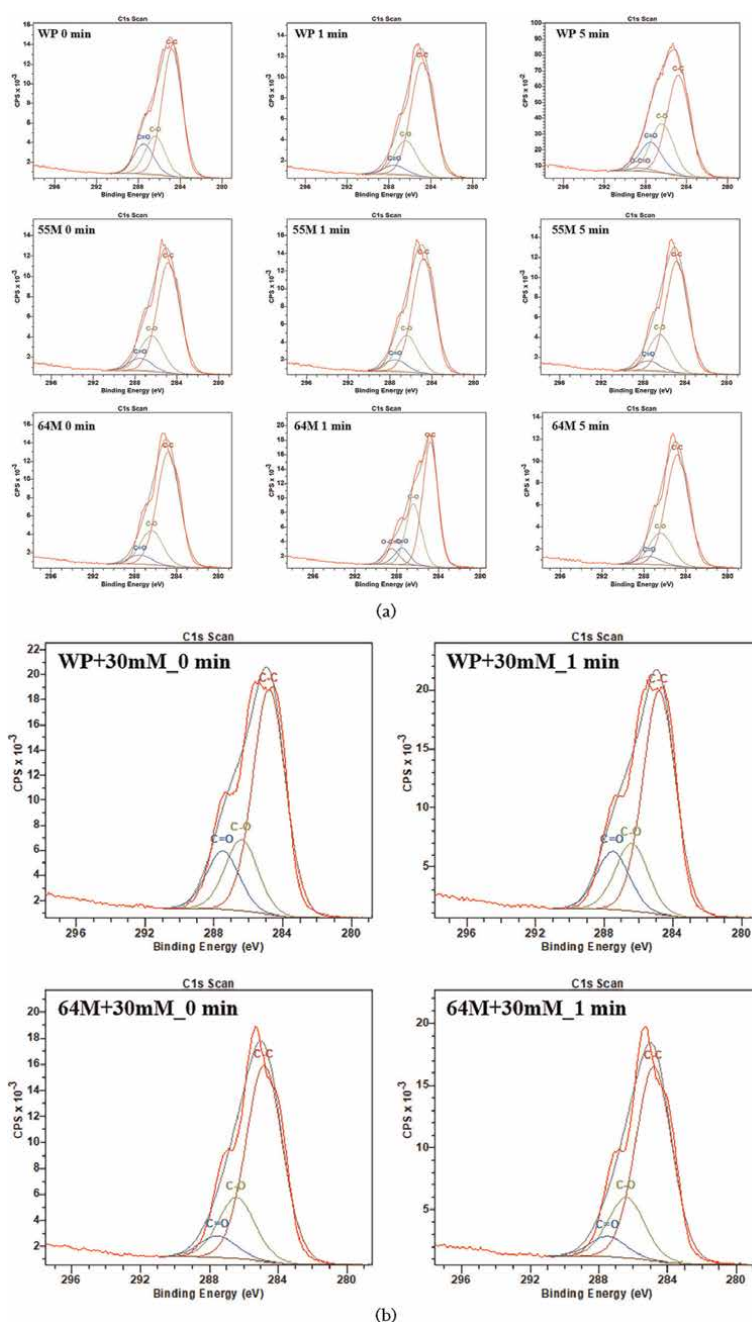
A. Appendix

See Table A1.

	WP0	WP1	WP5	55 M0	55 M1	55 M5	64 M0	64 M1	64 M5	WP 30-0	WP 30-1	64 30-0	64 30-1
C _{1s} (%)	65.68	64.26	64.40	63.42	63.79	62.42	62.85	69.07	62.87	66.04	65.59	63.66	63.04
O _{1s} (%)	24.13	28.05	25.85	27.84	27.34	28.17	28.42	22.65	28.03	24.79	24.30	27.76	28.83
N _{1s} (%)	10.19	7.70	9.75	8.74	8.87	9.41	8.73	8.28	9.10	9.17	10.11	8.58	8.13
Lactose (%)	19.35	34.28	24.88	32.07	30.46	31.43	33.72	18.17	32.04	22.80	19.95	32.09	35.81
Protein (%)	75.48	57.04	72.22	64.74	65.70	68.57	64.67	61.33	67.41	67.93	74.89	63.56	60.22
Fat (%)	5.17	8.69	2.90	3.19	3.84	nd	1.62	20.50	0.55	9.27	5.16	4.35	3.97
Calculation based on [6, 8, 31].													

Table A1.
Relative elemental surface composition of spray-dried milk powders.

B. Appendix



Author details

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