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Innovations in spray drying technology for liquid food processing: Design, mechanisms, and potential for application

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ABSTRACT

Spray drying is a well-established approach to converting liquid food into powder. This review discusses four types of spray drying techniques currently being used for that purpose, namely: pulse combustion (PC) spray drying, electrostatic spray drying (ESD), nano spray drying (NSD), and extrusion porosification (EP). PC spray drying is still in the development phase in the food sector, having the disadvantage of lacking industrial technology for heat-sensitive foods like milk or whey protein concentrates. On the other hand, it is able to reduce or even eliminate the viscosity limitations of atomizers, in addition to being considered a potential energy-saving option compared to conventional spray drying. ESD and NSD show promise in drying bioactive or heat-sensitive compounds like enzymes, cells, and vitamins. A nano spray dryer contains a spray mesh technology and electrostatic collector, which results in variations in particle size and product yield compared to traditional spray drying. However, the current types of equipment are only available on a laboratory scale. Also, NSD could be unfavorable when considering the low concentrations of feed solutions (0.1–1 % w/v). Finally, EP is claimed to provide better physical powder characteristics and permit the drying of highly concentrated liquids. It is hoped that these additional features and technologies will be further investigated in food systems to enhance process performance and improve powder properties.

1. Introduction

Drying is a well-established approach to preserve liquid foods, as it considerably limits microbial growth and enzymatic degradation reactions. Furthermore, the powdered form of food has advantages in transportation, distribution, and storage procedures due to the reduction in the product's final volume (Al-Hilphy et al., 2020). Additionally, the production of a powdered liquid favors the versatility in its application, both at the household and industrial levels.

Factors to consider when choosing a food-drying system include energy costs and sensory, nutritional, or technological alterations to the food matrix. If the drying process is incomplete or inappropriate, the quality of the final product can be affected. This implies unexpected changes, such as loss of food taste, color, or distinct moisture gradients, which can lead to an unmarketable product (Llavata et al., 2020). Therefore, several food-processing technologies have been developed that aim for better drying performance (lower operating costs and higher yields) and improved particle properties (e.g., particle size, color, rehydration capacity, flow properties, and bulk density). Most

techniques require significant energy input in the form of heat, such as conduction, convection, radiation, dielectric (microwave and RF), and fluidized bed (Mousakhani-Ganjeh et al., 2021). In acoustic drying, ultrasonic waves intensify the mass transfer, and ultrasonic oscillations elevate the temperature (Laborde et al., 2018). Conversely, osmotic drying is based on a mass transfer driving force, which takes place through the osmotic pressure difference by using a semi-permeable membrane.

Among the various systems for drying liquid foods, atomization by spray drying is the most common. In addition to being used for dehydration, it is widely employed in the encapsulation of essential oils (Campelo et al., 2017, Nguyen et al., 2022) and of probiotics in food matrices (Dantas et al., 2021; de Liz et al., 2020). In this system, the liquid formulation (solution, suspension, or emulsion [aqueous or organic]) to be dried is injected by a pump and dispersed at the entrance of a cylindrical container called a drying chamber. This dispersion (or atomization) occurs using a spray nozzle that converts the liquid into tiny drops. These droplets come into contact with the inlet gas (hot gas), resulting in the mass and heat transfer phenomena responsible for liquid

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evaporation.

Studies have found that since spray drying is a thermal process, it can induce negative effects on food. For instance, protein denaturation (Anandharamakrishnan, Rielly, & Stapley, 2007), loss of bioactivity of biological compounds (vitamins, enzymes, living bacteria) (Schutyser et al., 2019), and browning due to Maillard reactions (Gómez-Narváez et al., 2023; Koca Erbay, & Kaymak-Ertekin, 2015; Park, Stout, & Drake 2016) have been reported. Furthermore, according to Coelho et al. (2022), high energy costs can also represent a disadvantage when choosing this drying method. Sobulska, Wawrzyniak, and Woo (2022) discussed that the theoretical energy consumption of a properly designed and operated hot-air dryer is approximately 2620 kJ/kgwater evaporated (i.e., similar to the energy required to evaporate water). However, in practical terms, typical industrial dryers can consume more than 4500 kJ/kgwater evaporated. In this context, Baker and McKenzie (2005) showed a survey that determined the energy consumption of spray dryers in food, chemical, and ceramic industries. The values varied from 3000 to 20,000 kJ/kg $_{\mbox{\scriptsize water}}$ evaporated, with an average of 4870 kJ/kgwater evaporated. Sobulska et al. (2022) stated that using superheated steam as a drying medium instead of air can help to minimize the drying energy demand. They mentioned a novel compression technology that would result in a considerable diminution of the specific energy consumption (540–720 kJ/kg $_{\rm water\ evaporated}$). The authors named this system as superheated steam spray dryer with energy recovery. However, it must be noted that these data are assumed by the authors and lack practical research on spray dryers, in addition to seeming somewhat unrealistic.

It has also been observed that the spray drying of fruit and vegetable juices can result in low yield, which is defined as the ratio of the mass of powder collected over the mass of total solids in the feed (Tontul & Topuz, 2017; Jayasundera, Adhikari, Adhikari, & Aldred, 2011). The main reason for small powder recoveries is related to the low glass transition temperatures of the constituents of fruit juices. This contributes to the appearance of stickiness, leading to operational problems. Therefore, an approach to overcome the problem is to add to the feed solution carrier materials with high glass transition temperatures, consequently increasing its glass transition. Moreover, mechanical

scraping of the dryer, introducing cold air from the bottom, and using low-temperature dehumidified air were mentioned as strategies to prevent or reduce the stickiness during spray drying and thus increase the product yield. In summary, the drying operation's success depends on several factors: the technology used, drying time, temperatures, and the composition of the food to be dehydrated. Given this, knowledge of the most suitable operating conditions for a system, as well as the use of raw material pre-treatments, can minimize the potential difficulties. Thereby, scientists have been searching for tools that facilitate the achievement of appropriate drying parameters, which, as stated by Lisboa, Duarte, and Cavalcanti-Mata (2018), are often based on trial-and-error methodologies. Due to this, Lisboa et al. (2018) aimed to develop a simplified model of the spray drying process using an industrial spray dryer to support activities of new food formulation. According to them, the developed model successfully describes all spray drying operations independently of the product nature, and can be easily implemented in any software. In addition to key operating parameters such as drying gas outlet temperature or feed flow rate, the model can estimate final particle size, drying kinetics, wet bulb temperature, dew point, and glass transition temperature.

Considering the above, this review presents modified spray drying techniques that have been tested in liquid food on both industrial and laboratory scales. Therefore, we have discussed drying technologies claimed to produce better final product quality or lower cost than the conventional method. The production of powders for specific applications was also addressed. Table 1 summarizes the main differences between the technologies and their characteristics.

2. Pulse combustion drying

Spray drying technology can be modified to intensify heat and mass transfer. The utilization of a pulsating gas flow as the drying medium characterizes a pulse combustion (PC)-drying system. The pulsating gas flow originates from a pulse combustion unit, wherein the combustion occurs intermittently (in pulses) instead of the more usual continuous combustion. Thus, the intensifications of heat and mass transfer rates

Table 1
Main features of the four modified drying techniques.

Considerations	PC spray drying	Electrostatic spray drying	Nano spray drying	Extrusion porosification
Technique	It consists of a spray drying technology associated with a pulse combustion unit to generate the gas flow. The intermittent combustion creates oscillations of pressure, velocity, and temperature.	This technique is similar to the spray drying with an electric charge connected to the whole system.	This technique is similar to spray- drying with the introduction of a spray mesh technology and an electrostatic particle collector.	Drying system integrated by 3 steps: (1) vacuum evaporation of the feed; (2) twin screw extrusion-aeration of the viscous product; and (3) drying in the spray tower.
Drying air temperature	Starting from 180 °C	38–90 °C (Jayaprakash et al., 2023)	Starting from 80 °C	Starting from 170 °C
Optimizing parameters	Gas temperature Gas velocity Pulsation frequency Initial droplet diameter Chamber dimensions Initial solid mass fraction	Drying outlet temperature Atomizing pressure Feed rate Applied voltage	Inlet temperature Drying gas flow rate Spray rate intensity Applied voltage Spray mesh size Vibration frequency	Not referenced yet.
Solution properties	Concentration Viscosity	Not referenced yet.	Feed composition Viscosity	Not referenced yet.
Limitations	Not yet industrial technology for heat- sensitive foods, such as milk or whey protein concentrates. Possible competition in energy savings if high-temperature heat pumps are used to heat the inlet air.	This technique needs more scientific exploration to understand its benefits.	Feed concentration used in nano spray drying is relatively low (0.1–1% w/v), which restricts the use for several purposes.	This technique needs more scientific evidence to understand its effects on the final product and its economic viability.
Process scalability	Commercially available	Commercially available	Not feasible	Not referenced yet.
Example of application	Wu et al. (2015) Drying of egg white Combustion gas temp.: 326.6 °C Gas temp. in the chamber bottom: 76.6 °C Feed speed: 0.6 kg/min	Masum et al. (2022) Drying of high oil load emulsions, milk, and heat sensitive biomaterials Inlet temp.: <100 °C Outlet temp.: <60 °C	Díaz et al. (2019) Encapsulation of oleoresin paprika Inlet temp.: 80 °C Air flow rate: 100 L/min Mesh size: 7 μ m	Bouvier et al. (2013) Drying of protein milk concentrate Inlet temp.: 170–220 °C Outlet temp.: 55–75 °C Real outlet temp. measured in the particles: 45–55 °C Residence time: 15–20 s

(with consequent accelerating drying rates) are due to the oscillatory nature of the momentum transfer, intrinsically correlated to the pulsating jets' high temperatures and velocity (Meng, de Jong, & Kudra, 2016).

Although a pulse combustion system can be used in fluidized bed dryers and flash dryers, this work focuses on its application in spray dryers. Fig. 1 shows the layout of the main elements of a vertical PC spray dryer. In a simplified way, the apparatus consists of a PC unit connected to a conventional drying chamber. Inside the combustion chamber, air from a rotary air valve is mixed with fuel (propane or natural gas), causing explosions and the formation of hot air (up to 1000 °C) that is pressurized to about 2 kPa above the combustion fan pressure (Rehkopf & Mirko, 2017; Pulse Combustion Systems, 2007). This extremely hot flue gas rushes down the tailpipe, where it is blended with diluent/quench air to achieve the desired material contact temperature. This mixture is then conducted toward the atomization orifice. The course of air, fuel, ignition, and diluent air (i.e., the pulse combustor's frequency) can occur at a rate of 80-110 Hz or even 50-200 Hz, depending on the execution form (Rehkopf & Mirko, 2017). Meanwhile, the feed liquid formulation is continuously pumped into the atomization orifice, where the hot pulsating gases dry the fluid instantaneously (in less than 1 s, according to the Pulse Combustion Systems, 2021). It is known that the residence time in a conventional spray dryer can reach 30 s. Finally, the resulting solid particles enter a drying chamber and are usually collected downstream of the cyclone, while exhaust gases escape into the atmosphere.

Traditional spray drying requires the feed solution to be atomized into the drying chamber. There are three mechanisms by which the atomization (or spraying) can be performed: pneumatic nozzles, pressure nozzles, and centrifugal disk atomizers. Conversely, PC dryers do not require mechanical atomizers/nozzles because they use gas-dynamic atomization (no shear), permitting concurrent atomizing and drying of the liquid (Kudra & Mujumdar, 2001). This feature also allows the use of feed solutions with high solid content and/or high viscosity, whose atomization is difficult in conventional spray drying.

2.1. Commercial development of PC dryers

The oldest relevant patent related to PC dryers was published in 1983

(Lookwood, 1983), when Raymond M. Lockwood, in name of Jetsonic Processes Ltd. (Los Altos, CA) invented a PC fluidizing dryer. This device was intended to dry food and other moist products, in an inert atmosphere. After that, Gray and Lindahl (1986) developed an improved device to PC dry particulate materials. Then, many patents were deposited in 1987 (Gray, 1987a,b; Gray & Lindahl, 1987a,b; Gray & Marguth, 1987; Thaler & Rubens, 1987), with a notable increase in interest over the last few years. Recent stand-out patents are from 2011 and 2013 (Grobler et al., 2011, 2013), with Pulse Combustion Systems as the current owner of the technology. The inventors proposed a fluid diode within the combustion chamber's air inlet passage. In addition to permitting normal airflow into the chamber, the fluid diode may prevent the backflow of heated combustion products through the air inlet passage.

To date, we have found suppliers, developers, and researchers engaged in providing and developing PC drying technology worldwide. Concerning supplier companies, we can mention Novadyne Limited, Ekonek–Drying Innovation, Heat Technology Inc. (HTI), Pulse Holdings LLC (Pulse Combustion Systems), and Glatt Ingenieurtechnik (GmbH). Additionally, relevant contributions were carried out in academia through the works by Kudra (2008), Meng et al. (2016), Wu and Liu (2002), Zbicinski (2002), Zbicinski, Benali, and Kudra (2002), Zbiciński, Kudra, and Liu (2014), Kudra and Mujumdar (2014), Wu et al. (2015), Tsotsas and Mujumdar (2014), and Pramudita and Tsotsas (2019). Pulse Combustion Systems has been performed studies with lipid-rich products, and algae drying for biofuel, food, and nutriceutical purposes (Pulse Combustion Systems, 2023). Finally, the Institute of Agrifood Research and Technology (IRTA), Spain, has been working on milk drying using an equipment prototype developed by Ekonek.

2.2. Application in food ingredients

Few scientific investigations have employed PC spray drying in food products. Among them, we can cite Wu et al. (2015), who obtained egg white powder (P-PC) and compared it with egg white powder from a conventional spray dryer (P-RD). The authors noted that P-PC showed superior surface characteristics, more homogeneous size distribution, and a smaller mean particle diameter (D50 $=20.15~\mu m~x~D50 = 54.74~\mu m$) than P-RD. They also observed that the protein denaturation was

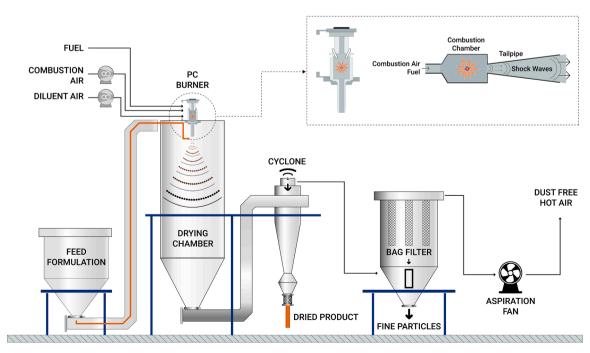


Fig. 1. Basic configuration of a pulse combustion spray dryer (Meng et al., 2016; Wang et al., 2007).

minor in P-PC, even when a hot drying gas temperature (326.6 °C at the feeding point) was used. Therefore, the PC spray drying process was assumed to protect the egg white proteins and their gelling and foaming properties. In addition, the energy efficiency (2604 kJ/kg water evaporated) was considered better than in traditional spray dryers (4500-11,500 kJ/kg water evaporated). Considering that the water evaporation latent heat is 2258 kJ/kg, the energy consumption calculated in this work seems implausible. The authors claimed that high energy efficiency is one of the distinctive merits of PC dryers, and similar results have been reported in other studies. Wu et al. (2015) still mentioned that the high energy efficiency can be correlated to the high-temperature and high-velocity oscillating gas produced by pulse combustion. These two factors would have enhanced the heat and mass transfer rates between the drying gas and feed solution (egg white). Likewise, Liu, Cao, and Lang (2001), who studied a Helmholtz pulse combustor, observed that the pressure amplitude and the oscillating frequency of the unsteady flow enhanced the convective heat transfer coefficient. According to Wu et al. (2015), the improved heat and mass transfer rates increased the drying rate and shortened the drying time of the egg white. The reasons associated with the increased drying rate were listed as follows: the smaller particle diameter of the powders; a highly turbulent flow field in the drying chamber; and the higher drying gas temperature (up to 326.6 °C, which is superior to those used in conventional spray dryer for sensitive food materials).

Pramudita et al. (2022) also studied the denaturation protein during PC drying using whey protein isolate (WPI), which is widely used as a wall material in encapsulation systems and as an ingredient in food products, presenting several properties: gelling agent, thickening agent, texture enhancer, surface-active agent, and foaming agent. WPI denaturation was investigated numerically by coupling a single droplet drying model with a kinetic denaturation model, followed by their experimental validations. For that, it was simulated the effects of gas temperature (80, 100, 120, 160, 200, and 300 °C), average gas velocity (3, 6, and 12 m/s), pulsation frequency (0, 40, 80, 120, 200, and 300 Hz), and initial droplet size (ranging from 10 to 130 μm with a 10 μm gap in between) on the process dynamic and quality product preservation. The results indicated that WPI smaller droplets ($D = 10 \mu m$) dried and denatured faster than larger droplets ($D = 130 \mu m$). Additionally, smaller droplets remained longer in the drying chamber (330 ms compared to 285 ms), given their ease of deceleration due to the oscillating gas velocity, unlike larger droplets with higher inertia. The average flow velocity had an insignificant effect on the properties of the dried product, but it strongly determined the droplet residence time (the higher the velocity, the shorter the time in the chamber). To prevent over-denaturation, the authors concluded that drying WPI slowly and at moderate gas temperatures (e.g., 120 °C) is better than drying it faster at gas temperatures of 140 and 150 °C. The drying process could also be intensified (while preserving the product quality) by employing a pulsation frequency in the gas flow of 80 Hz, which was strategically better than increasing its temperature, in addition to leading to lower energy consumption. Nevertheless, Pramudita et al. (2022) observed that the product properties vary depending on the process conditions. Moreover, the process parameters themselves can be interconnected in a complex way. For example, pulsation frequency changes dynamically with velocity and is a function of drying chamber dimensions and shape. This means that optimal conditions must be set case by case, taking into account specific product requirements and available technology.

Pramudita et al. (2021) conducted essays with maltodextrin and other components (SiO_2 and TiO_2) to analyze how the process conditions and material kinds influence the physical properties of the products. The PC spray dried-maltodextrin particles obtained by Pramudita et al. (2021) were mainly hollow with high porosity. A spherical shape was observed in most particles (94.7% by volume) from the condition at 400 °C (inlet gas temperature) and 80 Hz (gas pulsation). This portion decreased to 53% at 250 °C, replaced by non-spherical particles with shriveled surfaces. Maltodextrin usually undergoes thermal degradation

at temperatures above 250 °C. Thus, the authors conjectured that an inlet temperature of 400 °C (and outlet temperature of 242 °C) permitted the temperature in the central region of the tube to be beyond 250 °C, thereby facilitating the degradation of maltodextrin components. In turn, thermal degradation derives particles with different surface appearances. Pramudita et al. (2021) also applied mathematical modelling to describe droplet-level drying phenomena in a PC spray drying system (Fig. 2) and highlighted that the results obtained with the model were qualitatively in agreement with the experimental data.

As examined above, there are few published articles in the literature presenting food or food ingredients as feed solutions in PC spray dryers. On the other hand, the companies *Ekonek – Drying Innovation* and *Pulse Combustion Systems* have reported various materials tested at their respective pilot-scale PC spray dryers. As examples, we can see dairy products (whey, cheese, milk, permeates), broccoli, meat, fish byproducts, and cyclodextrin complexes. Likewise, pomaces and okara were cited as by-products that may be valued by PC drying and consequently, used as new ingredients for human food (Pulse Combustion Systems, 2023; Ekonek, 2023). However, it should be noted that these data lack scientific support, therefore being one of the avenues for future investigations.

3. Electrostatic spray drying (ESD)

ESD is a drying technique related to the electric field, which is considered a promising factor for the increased stability and encapsulation of dried particles. ESD is analogous to spray drying in that it comprises a closed layout and uses heating temperatures (however, lower than conventional spray drying, e.g., inlet temperatures < 90 °C) (Jayaprakash et al., 2023). The basic set-up of ESD technology consists of an electrostatic nozzle inside the drying chamber (at the top, lid), through which the liquid passes as it is downward-oriented and immediately atomized (Fig. 3). The material pumped through the nozzle is heated by pressurized gas and submitted to electrostatic actions. Consequently, static electricity forces the droplet's water toward its edge while the solid constituents remain in the center. This produces dried particles that are conducted to the separation plenum in a spiral flow pattern; these charged dried particles are collected and neutralized in a collector.

ESD technology has been primarily used in the field of bioactive compound encapsulation. This is due to the lower temperatures necessary to evaporate water in ESD systems. Moreover, it is supposed that the electrostatic interaction between the components during atomization also provides advantages for labile and heat-sensitive products. Nevertheless, it must be noted that ESD is a relatively new technology, with few studies exploring its benefits. More research is necessary to fill the current gaps in our knowledge of ESD, such as the influence of feed solution parameters on drying. Despite its novelty, ESD equipment is available. To the best of our knowledge, the only company commercially producing electrostatic spray dryers is Fluid Air (Naperville, IL, USA), a division of Spraying Systems Co., using a technology named PolarDry®. Substantial patents related to ESD are listed in Table 2.

One of the first works developed on ESD was performed by Johnson et al. (1996), using concentrated whole milk. Employing voltages of 0, 0.3, 0.6, 0.75, and 0.9 kV and atomizing air pressures of 140, 280, and 410 kPa, they found that the charging voltage had a significant effect on the solubility index of the dried sample. They also discovered that charging increased the particle diameter of the dried milk. Nonetheless, SEM and particle size analysis revealed that 140 kPa was insufficient for complete atomization.

Regarding thermosensitive ingredients, we refer to the studies by Wang et al. (2022) and Jiang et al. (2021), who worked on spray drying of Pu-erh tea and bifidobacteria microencapsulation, respectively. Specifically, Wang et al. (2022) applied ESD and other drying technologies to produce an instant Pu-erh tea, which is a special microbial post-fermented tea from *Camellia sinensis* var. *assamica*. In their work, it

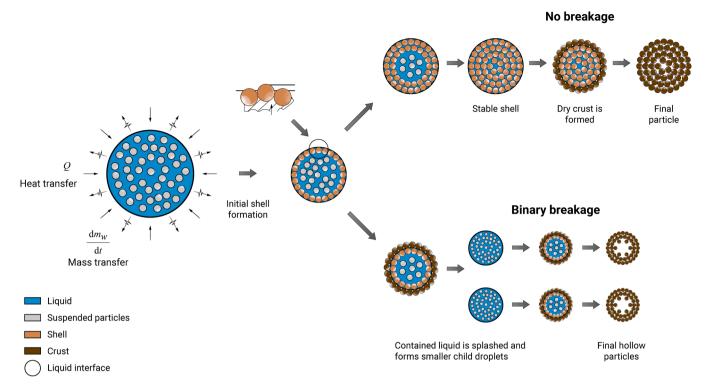


Fig. 2. Shell formation on the droplet surface (Mezhericher et al., 2011); and subsequent drying stages according to the hypothesis raised by Pramudita et al. (2021). They hypothesized that the breakage of the dry crust during the second drying stage is the phenomenon that justifies the existence of ultrafine particles in the final product. In this case, two main situations can explain the powder obtained: the successive breakage events that can occur (resulting in a variable number of child droplets); and: while the crust is split into two parts, a piece at the crack surface is fragmented in debris of fine particles.

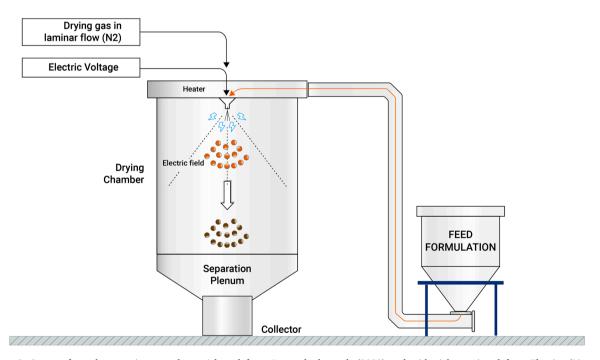


Fig. 3. Figure 3: Set-up of an electrostatic spray dryer. Adapted from Jayaprakash et al. (2023) and with rights assigned from Elsevier (License number: 5,486,521,217,735).

was observed that the sensory qualities (evaluated by a descriptive analysis) of electrostatic-dried tea were similar to those of freeze-dried tea. However, these characteristics were better than those of dried tea from vacuum drying or traditional spray drying. Namely, the intensities of fruity, sweet, stale, wood, and floral attributes were significantly

higher in the electrostatic-dried sample than in the spray-dried sample. Likewise, the number of odor-active compounds with an odor intensity ≥ 5 was higher in the electrostatic-dried tea than in the spray-dried tea (17 compounds versus 7 compounds, respectively). On the other hand, ESD was slightly less effective in preserving these components than

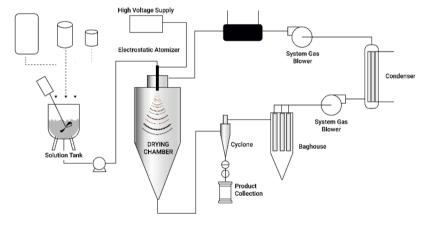
freeze-drying (17 compounds versus 24 compounds, respectively). However, freeze-drying is energy-intensive and expensive due to the prolonged drying time and the need for vacuum and freezing systems.

An advantageous employment of ESD was reported by Jiang et al. (2021), who found that *Bifidobacterium lactis* BL03 was better microencapsulated using ESD than using conventional spray drying or freeze-drying methods. The parameters investigated included survival and embedding rates post-encapsulation and survival rates after exposure to simulated gastrointestinal conditions. The survival rate after the

encapsulation process was 89.26% for ESD-encapsulated bacteria. Other studies on bifidobacteria microencapsulation showed survival rate values between 70.58 and 75.68% when spray drying was used (Fritzen-Freire et al., 2013; Simpson et al., 2005). After storage of 12 months at a temperature of 25 °C, Jiang et al. (2021) obtained a survival rate of over 70%, which is remarkably higher than those reported by Verruck et al. (2019), who found that spray-dried microencapsulated cells stored at 25 °C for 120 days had viabilities ranging from approximately 30% to 57%, depending on the carrier agent utilized. Furthermore, the survival

Table 2
Relevant patents on electrostatic spray drying.

Illustration	Patent (code, name, and assignee)	Brief information (purpose/set-up features)	References
	DE3630577A1 Electrostatic spray- drying method Bayer AG	The feed solution in a volatile solvent (methylene chloride) is atomized under the action of a strong electric field (range from 5×10^6 to 5×10^7 ohm cm). The dried solid particles (size between 8 and 40 μ m) are deposited on a counter-electrode.	Simm and Raue (1986)



US-8,939,388-B1 Methods and apparatus for low heat spray drying Zoom Essence Inc

was used as an integrative part of an encapsulation system by spray drying, in which the use of any heat current is avoided. For this purpose, a slurry was initially prepared and contained a carrier, a liquid solvent, and an active ingredient. Then, the slurry was submitted to the electrostatic charge, followed by atomization. Electrostatically charged particles still wet were obtained from this step and passed to the next until they reached dehydration. According to the technology assignee, this system improves the volatile flavor retention and decreases the loss to the atmosphere, contributing to a high-quality spraydried powder. The system allows the reduction of inlet and outlet temperatures, respectively: 25 °C to 110 °C 25 °C to 80 °C

It was designed to encapsulate core materials, such as volatile

flavor oil.

The electrostatic charge

Beetz et al. (2015)

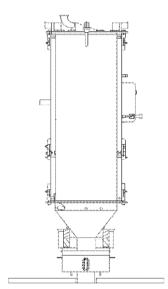
Sobel et al. (2016)

WO2016123224A1
Flavor encapsulation
using electrostatic
atomization Fona
Technologies Inc (US)

(continued on next page)

Table 2 (continued)

Illustration	Patent (code, name, and assignee)	Brief information (purpose/set-up features)	References
Comporte seed Cast Literal L	US20200171517A1 Electrostatic spray drying nozzle assembly Spraying Systems Co.	The work focused on the innovation of the electrostatic spray nozzle to produce a controllable fine liquid droplet spray at relatively high flow rates since this characteristic is advantageous in drying. The authors also developed an external mix electrostatic spray nozzle, which possibilities spray dryers to have more compact and shorter drying chambers.	Ackerman et al. (2020)



WO2021102231A1 Electrostatic spray dried milk product and production method thereof Spraying Systems Co. Equipment specially designed for powder milk production, where the application of the voltage is continuous (about 0.1 kV) at inlet temperatures below 150 °C. The resulting powdered milk presents agglomerate sizes of 100 pm or more, with a minimum of 10% carbohydrates in its composition and at least 8% less fat than the milk powder from a conventional spray dryer. Bogdan et al. (2021)

US20220105485A1 Flavor encapsulation using electrostatic atomization Fona Technologies Inc (US) The process contains the following steps: forming of an emulsion with at least one core component; dispersion of the emulsion in an electrostatic spray dryer to form droplets, applying an electrostatic charge of approximately 0.5 kV to 60 kV; and drying of the droplets at the same temperature conditions early

Sobel et al. (2022)

(continued on next page)

Table 2 (continued)

Illustration	Patent (code, name, and assignee)	Brief information (purpose/set-up features)	References
		commented. The core material can include any natural or created oil-based flavor, for example, spice, citrus, mint, essential oils, tropical fruit, or savory types. In addition, individual components can also be encapsulated: isoamyl acetate, benzaldehyde, ethyl butyrate, methyl salicylate, limonene, and menthol.	

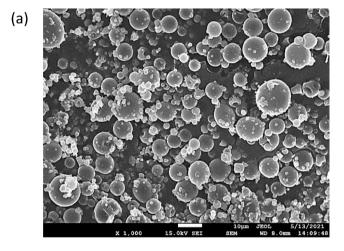
rate of ESD-encapsulated bacteria after 4 hours under simulated intestinal conditions reached 75%. These findings pave the way for several possibilities in the food industry due to the advantages of the cell powdered form, such as longer storage, versatility in application, and optimized delivery in the colon. Jiang et al. (2021) also showed that ESD had an improved effect on bifidobacteria microencapsulation based on SEM pictures. Additionally, a study more complete by Jiang et al. (2022) revealed that Bifidobacterium (6 strains of 3 species) dried using ESD had a better ability to relieve constipation in mice than that dried using conventional spray drying or emulsification freeze-drying. This improvement was attributed to enhanced maintaining of cell activity, changes in the composition of the intestinal microbiota, and subsequently, an increase in fecal water content and propulsion rate of the small intestine. It is important to note that the specific mechanisms responsible for superior preservation of cell viability and sensitive components, as reported by Wang et al. (2022), have not been sufficiently clarified. Nevertheless, the use of low inlet and outlet air temperatures, concretely 70 °C and 38-42 °C respectively, according to Wang et al. (2022), and an inlet temperature of 80 °C as set by Jiang et al. (2021, 2022), are undoubtedly contributing factors.

4. Nano spray drying (NSD)

For specific applications such as the production of nanoencapsulated food bioactive ingredients, smaller particle sizes are required, which is why this new version of the spray dryer, the nano spray dryer, has been developed. The principle of this technology is not fundamentally different from classical spray drying. The process consists of the following basic steps: heating of drying gas, droplet formation, droplet drying, and particle collection. Notwithstanding, some modifications to the experimental setup of a traditional dryer are necessary: the nozzle mechanism has to be able to generate smaller droplets; the drying gas flow needs to be laminar, not turbulent, and co-current with the pulverized droplets; and the particle collector needs to be particularly effective in separating submicron particles (Jafari, Arpagus, Cerqueira, & Samborska, 2021). As for the small droplet production, the atomization is carried out using a nozzle with vibration mesh at an ultrasonic frequency, which allows the obtaining of fine and uniform-size droplets. This spray mesh has an array of laser-drilled holes in the center (about 1500 holes in a 5.5 µm spray mesh), and its vibration is due to a piezoelectric actuator at a range of 80-140 kHz (Jayaprakash et al., 2023). These high-frequency vibrations cause the mesh holding feed to eject millions of droplets. Each hole functions as a micropump, drawing the fluid through the holes to produce identical droplets. These droplets form a thin, low-velocity aerosol (Chopde et al., 2020). For instance, with 1000 holes in the spray mesh and a frequency of 100 kHz, the atomizer generates approximately 100 million droplets per second. The uniformity of the droplets depends on the holes in the spray mesh,

resulting in dried particles ranging from 100 nm to 5 μm (Arpagaus et al., 2017).

As previously commented, the drying gas flow needs to be laminar. This is important since the turbulence would carry to an uncontrolled spray formation and particle deposits on the drying chamber sidewalls. Thus, the heating unit in the upper side of the nano spray dryer consists of a porous metal surface and creates a laminarization of the gas drying



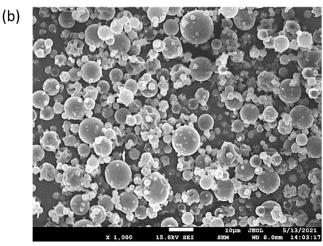


Fig. 4. SEM micrographs of oregano essential oil nanoparticles using different wall material ratios (whey protein isolate:maltodextrin): 1:1 (a), and 1:3 (b). Reprinted from Plati et al. (2021), under the terms and conditions of the Creative Commons Attribution (CC BY) license.

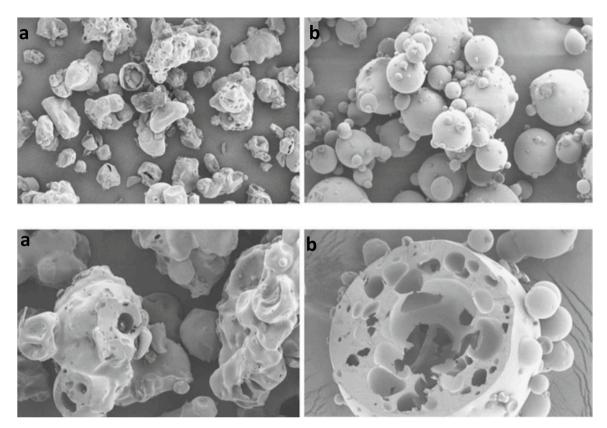


Fig. 5. The structure of spray-dried powder (a) was distinct from extrusion porosification powder (b). Spray-dried particles were irregular with a rough surface, had large internal vacuoles, and no surface pores; while extrusion porosification particles were spherical with a smooth surface and numerous pores, both inside and on the surface. Reproduced from Bouvier et al. (2013).

stream, with the Reynolds number varying between 330 to 660 at 40% humid air and 120 °C inlet temperature (Jafari et al., 2021). Finally, another crucial point of the technology is the electrostatic particle collector (placed at the bottom of the drying chamber), which is compounded by a smooth stainless steel cylinder (anode = particle collection electrode) and a star-shaped counter electrode (cathode) inside the cylinder. The achieved voltage between the two electrodes can be up to 20 kV, leading to an electric charging of the dried particles and their subsequent motion toward the inner wall of the collecting electrode. Unlike cyclones, the electrostatic charging is not dependent on the particle mass. This system allows for a particle recovery rate (yield) of up to 90% (BÜCHI, 2016).

The conventional spray drying system does not allow the achievement of nano-size particles because the cyclone vessel has limitations. Jafari et al. (2021) observed that typical cyclones cannot collect particles smaller than 2 µm, and even with high-performance models, the average particle size will still be above 1.4 µm. Additionally, the turbulent gas flow in the drying chamber of traditional atomizers can cause more particle deposition on the chamber wall. The higher yields are also one of the advantages inherent to the nano spray dryer of laboratory scale, permitting the atomization of very small amounts of sample (2 mL). The standard version of the equipment requires a minimum volume of 30 mL for a feasible test. This is made possible by the vibrating mesh's ability to aerosolize less quantity of fluids in the milliliter range (Chopde et al., 2020). Thus, the needed volume of liquid for the mini spray dryer corresponds to a few milligrams of solid, a relevant characteristic when working with expensive products of high-added value in the development phase.

As for the commercial development of the technology and its availability on the market, only two variants of the company BÜCHI Labortechnik AG (Switzerland) are available, both of laboratory scale (BÜCHI, 2023). The Nano Spray Dryer B-90 HP has been designed to spray-dry

aqueous solutions, nanoemulsions, or nanosuspensions, and the Nano Spray Dryer B-90 HP Advanced can additionally process organic-based substances.

NSD has been tested in several studies, many of which focused on the drying of proteins or peptides such as bovine serum albumin (BÜCHI, 2017; Lee et al., 2011; Pedrozo et al., 2020), enzymes (Bürki et al., 2011; Abdel-Mageed et al., 2019, 2021), and lactoferrin (Bourbon et al., 2020). Nanoencapsulation of food compounds in whey protein wall systems was also studied (Prasad Reddy, Padma Ishwarya, & Anandharamakrishnan, 2019; Pérez-Masiá et al., 2015). The drying of proteins and peptides is relevant because they are highly affected by external factors during processing and storage, and depending on their intrinsic characteristics, they are also easily altered by deteriorating microorganisms (Jafari et al., 2021). Thereby, the solid form of proteins and peptides provides better storage stability. In addition, Haggag and Faheem (2015) cited some advantages of NSD against spray drying in protein dehydration. For instance, in the context of proteomics and genomics aimed at pharmaceutical applications, nanosized powder enhances the stability and therapeutic potential of the drugs, making them less toxic and more effective. The size of the drugs significantly affects their biodistribution and clearance in vivo. Moreover, Bourbon et al. (2020) investigated the NSD of protein-based nanohydrogels and proved that the process did not affect their structure and morphology. They used lactoferrin (an iron-binding glycoprotein found in several biological mammal fluids) and glycomacropeptide (glycosylated phosphate peptide derived from κ -casein that has attracted attention due to its bioactive properties) for the formulation of the nanohydrogels. The authors also verified that the dried particles obtained were more spherical when contrasted with those from the freeze-drying process.

NSD was equally applied in works with essential and functional oils (Brinkmann-Trettenes et al., 2013; Hu et al., 2016; Wang, Soyama, & Luo, 2016; Veneranda et al., 2018). Plati, Papi, and Paraskevopoulou

(2021) observed high levels of powder recovery (>77%) for oregano essential oil encapsulated in whey protein isolate-maltodextrin mixtures. Their study also showed that the particles produced were spherical and very fine (see Fig. 4). Furthermore, the nanocapsule's activities were stronger than pure oregano essential oil against *Staphylococcus aureus* and *Escherichia coli*.

5. Extrusion porosification (EP)

Clextral, a company with expertise in extrusion processes, developed a technique named Extrusion Porosification Technology. It consists of a drying system integrated by 3 steps: (1) vacuum evaporation of the feed until it reaches viscosities of 2–5 Pa·s (with the possibility of work up to 20 Pa·s); (2) twin screw extrusion-aeration of the viscous product, conducted in the presence of gas (typically CO_2 or N_2); and (3) drying, wherein the high solids textured foam produced in the previous step is injected into the spray tower. During expansion, the gas dissolved in the viscous liquid is vaporized, removing water molecules. Concomitantly, a porous (honeycomb) structure is formed in the foam, and then, in the powder (Fig. 5). This particle shape leads to faster moisture transfer during drying, allowing the outlet temperature of the product to be reduced significantly (Brisset & Collado, 2016; McHugh & Maller, 2019).

One of the claims about EP is that this technique can be used to dry highly concentrated liquids. That is, it could handle much more viscous materials than regular spray drying (Clextral, 2023). However, there is a need for more scientific results to support this claim, as the method was tested in only one published work (Bouvier et al., 2013). This study showed that milk protein concentrate powder from traditional spray drying had a lower dispersibility index (38%) than extrusion-porosified milk protein concentrate powder (96%) after exposure to the same conditions. The authors attributed it to the dissociation of casein micelles induced by the extrusion porosification treatment preceding drying. Changes in the microstructure of extrusion-porosified powder particles were proposed to support the expressive improvement of the rehydration behavior.

6. Conclusion and prospects

Four major areas of emerging technologies were reviewed: PC spray drying, ESD, NSD, and EP spray drying. PC technology is able to reduce or even eliminate the viscosity limitation of atomizers, which is why it has been tested in several foods, by-products, and pastes. ESD has mainly been used to encapsulate sensitive or thermolabile components. Low work temperatures and the electric charge are its distinguishing features. Commercial equipment for ESD is already available, but the technique requires further investigation before it can be considered for widespread use. NSD is similar to spray drying, with a vibrating spray mesh for droplet generation and an electrostatic particle collector that possibilities an optimized collection of nanoparticles. Since conventional spray drying is limited regarding particle size, NSD technology has emerged recently so that powder nanoparticles with a narrow size distribution can be produced effectively. However, the current nano spray dryers are only available on a laboratory scale, making it difficult to commercialize relevant food products. Thus, more research is needed in this area, as pilot-scale or even industrial-scale nano spray dryer development would be very useful to produce fine powders with nano traits, which is undoubtedly one of the modern demands. In turn, EP (extrusion porosification) is claimed to provide better physical powder characteristics and permit the drying of highly concentrated liquids. Nonetheless, there is a need for more scientific results to support this claim. Finally, it is hoped that these additional features and technologies will be further investigated in food systems to enhance process performance and improve powder properties.

CRediT authorship contribution statement

Adriana Dantas: Conceptualization, Investigation, Visualization, Writing – original draft, Writing – review & editing, Methodology. Marc Piella-Rifà: Investigation, Visualization, Writing – original draft. Diogo Pontes Costa: Conceptualization, Investigation, Writing – review & editing. Xavier Felipe: Funding acquisition, Project administration, Supervision, Writing – review & editing. Pere Gou: Conceptualization, Investigation, Project administration, Supervision, Writing – original draft, Writing – review & editing.

Declaration of competing interest

None (there are no interests to declare).

Data availability

No data was used for the research described in the article.

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