

**NANO- AND MICRO- ENCAPSULATION OF CURCUMIN BY SPRAY
DRYING TECHNIQUE: CHARACTERISTICS AND APPLICATIONS IN
FOOD**

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Abstract

Curcumin (CUR) is an important natural polyphenol, extracted from the rhizome of *Curcuma Longa* used for thousands of years by the Asian population, both as a food ingredient (dye, preservative, spice) and as a remedy for certain gastrointestinal diseases, inflammation, cataracts, and bacterial infections. Unfortunately, CUR, like many polyphenols, has a low bioavailability due to its low water solubility, chemical instability at pH variations, low cell absorption, and rapid metabolism. To overcome these challenges, many researchers have applied various CUR encapsulation techniques. In this study, an overview of the principles and conditions for nano/micro-encapsulation of CUR through the spray-drying has been presented. Different biopolymers including polysaccharides and proteins have been utilized for this purpose. For nanoencapsulation of CUR, an innovative spray-drying technique has recently been developed, the so-called nano-spray-dryer, whose operations and advantages are briefly discussed in this paper too. The results from the literature on the optimization of physico-chemical and technological parameters, the nature of encapsulating materials, and the characteristics of the spray-dried CUR-loaded powder particles are also highlighted. Finally, the applications of spray-dried CUR powders in food functionalization with the focusing on the antioxidant, antimicrobial, and coloring properties are described.

Keywords: curcumin, spray drying, powder properties, nanocarriers, functional foods

Introduction

Today, more than ever, feeding the population has become a major problem for the whole world. The agri-food sector is called upon to ensure, on the one hand, the minimum food requirement for a worryingly large number of people suffering from hunger, and on the other hand, to focus on increasing food quality and safety, in order to contribute to the health and well-being of consumers.

Also, food researchers together with food producers have adopted new strategies to develop innovative processing technologies that enable the design and development of innovative foods capable of preventing and treating diseases (De Vries *et al.*, 2018; McClements, 2019). In this regard, specialists from various fields, such as food nanomaterials, food science and engineering, medicine, nutrition, and pharmacy, have developed new foods, with an important role in preventing and treating diseases, such as functional foods and medical foods to which nutraceuticals are added (McClements, 2019).

The production of functional foods and nutraceuticals is based both on the experience and millennial tradition of humans in using natural resources (plants, minerals, animals) to treat diseases, as well as on the results of scientific research on the biological activity of phytochemicals, microorganisms, and various extracts from animal organs and tissues (Granato *et al.*, 2020).

A wide range of bioactives and nutraceuticals are used in the preparation of functional foods, including vitamins, minerals, prebiotics, probiotics, hydrocolloids, essential oils, omega-3/6 fatty acids, polyphenols, and carotenoids (Martirosyan *et al.*, 2022). They are included in various food matrices, such as bakery, cereals, dairy products, meat, fish, eggs, soy products, intended for the diet of special groups of consumers (athletes, infants, young, old) in order to ensure optimal nutrition and intestinal prevention disorders (ulcers, microbial, viral, and parasitic infections), coronary heart disease, high blood pressure, high cholesterol level, high blood sugar level, high triacylglycerol level, inflammation, cancers, etc. (Ghani *et al.*, 2019; Dima *et al.*, 2023).

Polyphenols are an important class of nutraceuticals including over 8000 phenolic compounds found in various fruits and vegetables (Di Lorenzo *et al.*, 2021). Recently, many foods and beverages have been fortified with plant extracts rich in polyphenols (Li *et al.*, 2023). *In vitro* and *in vivo* studies have highlighted the complex biological potential of polyphenols, whose antioxidant activity (AOXac) has been extensively analyzed (Dienaite *et al.* 2019; Stagos, 2020). Thus, polyphenols, as antioxidant agents, inhibit the oxidative degradation of lipids, proteins, and vitamins in food (Papuc *et al.*, 2017; Nazzaro *et al.*, 2022). The high ability of polyphenols to capture free radicals explains their involvement in reducing oxidative stress, which seems to be the main cause of cardiovascular disease, cancer, neurodegenerative diseases, etc. (Cory, *et al.*, 2018; Almeida *et al.*, 2022). Recent studies have highlighted the role of polyphenols in the prevention and treatment of other diseases, e.g., osteoporosis, obesity, type 2 diabetes, gastrointestinal disorders, and inflammations (Boaru *et al.*, 2024; Zhao *et al.*, 2024).

Curcumin (CUR) is an important natural polyphenol, extracted from the rhizome of *Curcuma Longa* used for thousands of years by the Asian population, both as a food ingredient (dye, preservative, spice) and as a remedy for certain gastrointestinal diseases, inflammation, cataracts, and bacterial infections (Jain, 2025). Recent studies have shown that CUR acts as a nutraceutical with a wide range of biological activities, such as: antioxidant, antimicrobial, anti-inflammatory, anti-diabetic, anti-tumor, and neuroprotective properties (Adiwidjaja *et al.*, 2017; Farhood *et al.*, 2019; Hartogh *et al.*, 2020; Hettiarachchi *et al.*, 2022). Unfortunately, CUR, like many polyphenols, has a low bioavailability due to its low water solubility, chemical instability at pH variations, low cell absorption, and rapid metabolism (Karthikeyan *et al.*, 2020).

The results reported in literature showed that CUR is a relatively hydrophobic molecule ($\log P \sim 3.2$), with poor water solubility (3.12 mg/L at 25°C), unstable in biological fluids ($\text{pH} > 7$), with a half-life of $T_{1/2} = 5\text{-}10$ min. (Martínez-Guerra *et al.*, 2019; Zheng and McClements, 2020). *In vitro* studies using the Caco-2 cells model also showed that the permeability coefficient of CUR is very low ($P_{\text{app}} = 3.18 \times 10^{-6}$ cm/s) which explains its very low bioavailability (<1%) (Faralli *et al.*, 2019; Purpura *et al.*, 2018). Scientists have investigated new strategies to improve the bioefficiency, sensory, nutritional, and biological effects of CUR. Some of these strategies are based on methods that reduce particle size (S_p), such as wet milling, ultrasonication, and solid dispersion, while others are based on the self-assembly of CUR molecules into nanoparticles (NPs) by nanoprecipitation, antisolvent precipitation, and solvent evaporation (Purpura *et al.*, 2018). Moreover, the nanoformulation of CUR can be achieved by its incorporation into different nanocarriers via emulsification, self-emulsification, coacervation, gelling, spray drying (SD), freeze drying, etc. (Cretu *et al.*, 2011; Silva *et al.*, 2018a; Kharat *et al.*, 2019; Dourado *et al.*, 2022; Goranova, 2024).

In this paper, an overview of the principles and conditions for obtaining encapsulated CUR and nanoCUR (nCUR) by SD method is made. The results from the literature on the optimization of physicochemical and technological parameters, the nature of encapsulating materials and the characteristics of the spray-dried CUR are also highlighted. Finally, the applications of spray-dried CUR powders in food functionalization with the focusing on the antioxidant, antimicrobial, and coloring properties are described.

Micro- and nano- spray-drying of curcumin

Spray drying is an important technique for preparing nCUR either in the form of pure CUR nanocrystals (CURNCs), coated with different materials, or in the form of nanoparticles (NPs) loaded with free or suspended CUR in various colloidal systems (nanoemulsions, double emulsions, microemulsions, liposomes, solid lipid nanoparticles (SLNs) (Wang *et al.*, 2018; Zheng *et al.*, 2018; Guo *et al.*, 2020; Opustilová *et al.*, 2024).

Spray drying is based on simple and versatile technologies, easy to handle, with high efficiency, and a low cost. Today, SD technique has become a preferred method of drug and food manufacturers, due to the increased performance of the machine and computerized optimization of SD processes and parameters to obtain particles with physicochemical characteristics specific to the field of application (Akhavan *et al.*, 2016; Guo *et al.*, 2020).

The conventional SD (cSD) has been extensively used for the encapsulation of bioactives, e.g., polyphenols (Ceglediet *et al.*, 2022; Tsatsop *et al.*, 2025), polyunsaturated oils (Sultana, 2023), essential oils (Phanse *et al.*, 2024), and vitamins (Bajaj *et al.*, 2021). For some applications, such as pharmaceuticals, particles with submicron size (100 - 1000 nm) are required. NPs improve the functional properties of bioactives (solubility, AOXac, and antimicrobial activity) and can be more easily absorbed through the intestinal epithelial layer, helping to increase the bioavailability of drugs and nutraceuticals. In this regard, Büchi Company in Switzerland designed, in 2009, a new generation of spray-dryer that produces NPs with narrow size distribution (Arpagauset *et al.*, 2018). In the last two decades, many specialists described and capitalized on the performance of nano-SD (nSD) in encapsulating bioactives, such as: CUR (Xue *et al.*, 2018; Boaru *et al.*, 2024), resveratrol (Consoli *et al.*, 2023), β -carotene (Lavanya, 2020), saffron extracts (Kyriakoudi and Tsimidou, 2018), and vitamins (Oliveira *et al.*, 2013; Singhet *et al.*, 2025). The SD technology has many benefits compared to other methods for preparing drug and functional food ingredients, but, unfortunately, it also has some drawbacks summarized in Table 1.

This section provides a comparative study of the technological characteristics and performance of two SD techniques: cSD and nSD for the manufacture of micro- and NPs. During SD, briefly, the liquid material is atomized into fine droplets which, in contact with a heated gas, instantly loses water by evaporation and turns into solid particles with minimal moisture (Arpagaus 2019).

Both cSD and nSD are based on the following main operations: infeed preparation, atomization, drying the feed droplets, separation and collection of particles. Each operation is carried out under the control of parameters whose variation crucially influences the characteristics of particles. Some of these operations differ in the two types of spray dryers due to the use of specific technologies for the preparation and collection of micrometric and submicron particles. These differences are highlighted in Figure 1 and Figure 2.

Infeed preparation

To improve the functional and biological properties of CUR, researchers have applied two important strategies, such as transforming native CUR, extracted from turmeric tubers, into nanoparticles, e.g. curcumin nanocrystals (CURNCs) and encapsulating CUR in different colloidal delivery systems obtained by different methods, of which the SD method proved to be the most efficient. The preparation of solid powders of CURNCs is done by physicochemical methods, including sonication, anti-solvent precipitation, and nano-suspension methods (Bonaccorso *et al.*, 2020). Moreover, CURNCs powder can be obtained by SD technique using CUR

suspensions without coating material, in the presence of surfactants that contribute to the formation of CURNCs inside the association micelles. For example, Hu *et al.* (2015) prepared CURNCs using a suspension formed by mixing CUR with an aqueous solution of Tween 80. The CUR suspension was first ground using yttrium-stabilized zirconium oxide beads (0.6 mm in diameter) as a grinding medium and then spray dried under inlet temperature (T_{inlet}) = 170°C, outlet temperature (T_{outlet}) = 90°C, and feed low rate (FFR) = 3 mL/min. The size of CURNCs decreased at higher grinding times. Thus, the average diameter of 50% of CURNCs (D_{50}) was 1277 nm after milling for 10 min, and 924 nm after milling for 40 min, respectively.

Table 1. Advantages and drawbacks of conventional spray drying technique (Assadpour and Jafari, 2019; Arpagaus, 2019).

Advantages
<ul style="list-style-type: none"> • converts liquid systems (solutions, suspensions, emulsions, pastes) into powders, easy to pack, transport and process • the powders produced by spray drying have a low moisture content, and consequently an increased shelf life • involves simple processes, and rapid operations with easy-to-handle installations • ensures solvent recovery and gas recirculation • allows for the modernization and computerization of operations such as the atomization of feed materials, the drying and collection of micro/nanopowders • allows for the control and optimization of parameters in order to make the drying process more efficient • allows for the control of thermal encapsulation conditions to avoid degradation of heat-sensitive bioactives • both the hydrophilic and lipophilic bioactives can be encapsulated • ensures the protection and stability of bioactives against oxidation, light, and temperature • stable particles with high encapsulation efficiency are obtained • can be applied at laboratory, pilot, and industrial scale
Drawbacks
<ul style="list-style-type: none"> • spray drying processes require high energy consumption • the drying yield is limited (50%-70%) by the tendency of powder to deposit on the drying chamber walls • the uniformity of microparticle size is affected due to powder agglomeration • high temperature and rapid vaporization process lead to the loss of volatile components from the liquid feed material • high-performance equipment of spray dryers (atomizers, gas heaters, powder collectors) and their maintenance increase the cost price of the spray drying technique • the spray drying process can affect the environment due to gas emissions and the release of microparticles into the air

Buchi Company also reported a protocol for the preparation of nCUR by SD of CUR solutions (0.1%) in ethanol, or in a mixture of ethanol and acetone (1:1) (www.buchi.com).

buchi.com.2017). For SD of the two solutions, Nano-Spray Dryer B-90 HP was used, and the control parameters were: nebulizer (mesh holl diameter 4 μm), air flow rate (AFR) (120 - 150 L/min), T_{inlet} (65 - 75°C), T_{outlet} (39 - 45°C), spray rate (80%), pressure (36 - 55 hPa) and feed rate (10%). The size of CURNPs was slightly different, depending on the solvents used. Thus, for CUR in ethanol solution, nanoparticles with size particle (Sp) of 0.367 - 1.29 μm were obtained, while for CUR in solvent mixture (ethanol/acetone 1:1), the Sp was between 0.428 - 0.974 μm .

For the encapsulation of CUR using the SD method, the feed material (FM) was prepared by mixing the wall material with free or pre-encapsulated CUR in different colloidal delivery systems (CDSs) including emulsions, microemulsions, double emulsions, SLNs, liposomes, etc. (Hu *et al.*, 2018; Preis *et al.*, 2019; Rafiee *et al.*, 2019). CUR is first solubilized in a lipid and then mixed with an aqueous suspension of wall material, forming emulsions. The emulsion stability is ensured either by the wall material, which can itself have emulsifying properties (gum Arabic (GA), proteins), or by adding synthetic emulsifiers (Tween 80, Tween 40, etc.). The emulsion droplet size is an important parameter that affects the drying process, encapsulation efficiency (EE) and loss of volatile components during SD, while the encapsulating material affects the viscosity of emulsion and ensures an optimal level of total solids content (TSC) for the FM. The TSC influences SD process differently. Thus, a high TSC increases the dryer flow rate, improves volatile retention, and increases the viscosity of the FM making atomization and drying more difficult. Moreover, a high viscosity FM causes the formation of large droplets that will dry insufficiently and stick to the dryer wall. The optimal TSC in FM is established depending on the nature of wall material, atomization technique, dryer configuration, and the required characteristics of the powders (Jafari *et al.*, 2021). In most papers, the optimum TSC reported was between 20 - 40% (Araga *et al.*, 2025).

Carrier materials used for encapsulating drugs and food ingredients must first be certified *Generally Recognized as Safe* (GRAS) and approved by government agencies, such as the U.S. Food and Drug Administration (US FDA), the European Food Safety Authority (EFSA), etc. The selection of the encapsulation material for the SD method is made in accordance, on the one hand, with the requirements of the technical operations and, on the other hand, with the physicochemical characteristics of the micro and NPs specific to the applications. It is also recommended that FM have high solubility in a certain solvent and low viscosity at high concentrations, emulsifying properties and form protective films against oxygen, light, and humidity, low hygroscopicity, high molecular weight, glass transition temperature (T_g), and degree of crystallinity to increase the retention of bioactive and reduce the agglomeration process of particles and their sticking to the wall of the drying chamber (DrC) or particle collector, not to react with the encapsulated bioactives, not to affect the taste and smell of the particles, not to affect the environment, and, last but not least, to optimize the cost/benefit ratio (Jafari *et al.*, 2008). Some materials should be pH sensitive for targeted release of bioactives (Lu *et al.*, 2021; Araga *et al.*, 2025).

The most common materials for encapsulation of CUR by SD are biopolymers, including natural biopolymers (carbohydrates and proteins), synthetic and modified biopolymers, and biopolymer mixtures.

Native carbohydrates, such as starch, cellulose, and chitosan, are less commonly used for encapsulation of hydrophobic compounds, due to their low emulsifying properties and low T_g which affect particle morphology and EE (Espíndola *et al.*, 2023). The preferred carbohydrates for encapsulating CUR by SD are hydrolyzed starches (HS), modified starches (MS), and some gums (Aniesrani *et al.*, 2015; Cano-Higueta *et al.*, 2015; Gałkowska *et al.*, 2023). HS is a mixture of polysaccharides obtained by acidic or enzymatic hydrolysis of native starch. The physicochemical properties of these mixtures depend on the hydrolysis degree evaluated using an empirical parameter, called Dextrose Equivalent (DE) (the amount of reducing sugars expressed in α -D-glucose (dextrose), existing in 100 g of dry matter). HS with $DE < 20$ are known as maltodextrins (MD), and those with $DE > 20$ are classified as corn syrups solid. MDs with $DE 5-10$ are generally used as wall material due to higher glass transition temperatures (T_g) than those with higher DE (Laurenti *et al.*, 2023). The low T_g reduces the retention of CUR and deteriorates the quality of powders due to sticking, agglomeration, and aggregation of particles. Extensive use of MDs as wall material is due to their special properties, e.g., high solubility ($> 70\%$), low viscosity at high TSC, protection against oxidation, retention of flavor, and low cost. Unfortunately, HS does not have emulsifying properties, and limits their use to the preparation of FM used to encapsulate hydrophobic compounds, such as CUR. To eliminate this shortcoming, the HS is mixed with MS or some gums, such as GA, which are good emulsifiers. MS with good emulsifying properties is obtained by chemical reaction of starch or HS with various compounds containing a hydrophobic chain. Thus, a starch with an amphiphilic structure is octenyl starch succinate obtained by the reaction between starch and octenyl succinic anhydride.

Gums are polysaccharides extracted from seeds, fruits, and other plant organs. GA is probably the most used natural biopolymer for encapsulating bioactives, by SD. GA or gum acacia, is an exudate extracted from the stems and branches of *Acacia* species, in particular from *Acacia Senegal*. Due to its amphiphilic structure, GA is a very good emulsifier used successfully in the preparation of FMs. It is soluble in water and forms concentrated solutions (50%) with low viscosity. Moreover, GA reduces crystallization, forms films, and is used as a surface-finishing agent (Rosland *et al.*, 2020). The protection capacity of encapsulated bioactives against oxidative degradation varies depending on the acacia species from which it was extracted. When mixed with MDs, GA improves the retention and controlled release of bioactives, contributes to higher T_g , and reduces hygroscopicity and particle agglomeration (Zhang *et al.*, 2019). The disadvantage of GA is the higher cost than the one of starch, because of its limited natural sources. Some authors have reported CUR encapsulation using GA alone or mixed with other materials (Bucurescu *et al.*, 2018; Zhang *et al.*, 2019; Meena *et al.*, 2021;).

For example, Lucas *et al.* (2020) published a comparative study on the influence of wall material on SD and the characteristics of CUR-loaded microparticles (MPs).

For this purpose, GA, sodium alginate, and modified chitosan, at 1% concentration were used. The FM was prepared by mixing the biopolymer solution with a 0.9% solution of CUR in ethanol. The solutions were spray-dried under T_{inlet} (150°C), AFR (40 m³/h), FFR (4 mL/min), and air pressure (6 bar). The yields of obtaining CUR-loaded powders were higher than for CUR-free powders, and the yield for obtaining CUR-loaded NPs with modified chitosan, alginate, and GA was 57.3, 54.5 and 46.1% respectively, while EE was 93.8, 97.0 and 97.6% respectively. Other gums were used to encapsulate CUR by SD such as inulin, alginate, agar, pectin, and tamarind gum (Table 2).

Proteins have excellent functional properties that allow for their successful use as wall material in SD. Thus, proteins are very good emulsifiers and stabilizers, form protective films, increase the EE and have the ability to bind minerals or some bioactives such as CUR (Li *et al.*, 2013). Milk/whey/soy proteins are used to encapsulate CUR. Some authors prepared powdered milk loaded with CUR, by SD, using skim milk concentrates mixed with an alcoholic solution of CUR (Neves *et al.*, 2019). SD was performed under the following technical conditions: T_{inlet} (161 - 165°C), T_{outlet} (88 - 92°C), air pressure (1bar), and FFR (350 - 500 mL/min). The drying yield of CUR-enriched powdered milk was (69 -85%), higher than the yield of CUR-free powdered milk (68 - 74%). Other authors encapsulated CUR in soy protein isolate (SPI) and studied the water redispersion behavior, bioaccessibility, and bioactivity of encapsulated CUR (Chen *et al.*, 2020). The addition of soy soluble polysaccharide (SSPS) and/or MD, alone or in a combination, to SPI, led to higher TSC, contributing to protection against CUR degradation during SD. Wang *et al.* (2017) also showed that adding MD in the wall material increased the retention of CUR in the spray-dried CUR-loaded octenylsuccinated corn dextrin micelles.

Milk and whey proteins have also been used to functionalize CUR by SD (Liu *et al.*, 2016; Kavousi *et al.*, 2018). To improve the solubility and stability of CUR, Khanji *et al.* (2018) encapsulated CUR in micellar caseins and then converted the liquid to powder by SD. The retention of CUR in powder was approximately 97%, and AOXac of CUR-loaded powder was 88% of that of free CUR. In fact, by SD of micellar caseins loaded with CUR, the stability of CUR was increased and AOXac was maintained for 60 days of storage at 40°C. Similar results were reported by Pan *et al.* (2014), who studied the dispersibility and bioactivity of CUR encapsulated in sodium caseinate micelles. The CUR powders obtained by SD were easily dispersed in water and showed a solubility 40 times higher than the solubility of pure CUR. Therefore, biological properties of encapsulated CUR, such as AOXac and cancer cell proliferation, were higher than those of free pure CUR. The use of proteins in the encapsulation of CUR is limited by the influence of pH which, affects both protein solubility and CUR stability.

Table 2. Overview of main operational conditions of spray-drying and characteristics of curcumin-loaded micro/nanoparticles.

Wall material	Spray-drying conditions	Particles size	Encapsulation efficiency (%)	Main results	Ref.
Conventional spray drying					
SA, GA, MCH	Spray nozzle: 0.5 mm; T _{inlet} : 150°C; FFR: 4 mL/min AFR: 40 m ³ /h; T _{outlet} : 68-70°C	Differential volume distribution: SA: 4.31 µm GA: 4.22 µm MCH: 41.55 µm	SA: 97.0 GA: 97.6 MCH: 93.8	CUR release kinetics and transport mechanisms were studied by Korsmeyer-Peppas and Weibull models; - SA MPs and MCH MPs showed diffusion-controlled release; - GA MPs showed diffusion- and swelling-controlled release; - Release rate: 63.2% after 60 min.	(Lucas et al., 2020)
Mixtures of : MS or IN, with: G, MD, β-CD, P, TG	Spray nozzle: 0.7 mm; T _{inlet} : 190°C; FFR: 600 mL/h;	10 -100 µm	41.36 (IN+βCD+P) – 82.5 (IN+MD+TG)	MC of CURMPs was between 1.9% and 10.1%; Color analysis: - L*: 79.58 – 93.62; - b*: 75.12 (mixture of IN, MD and P); 91.48 (mixture of MS, βCD andP); CUR acid stability (R %): 80.7 (mixture IN, MD and G); 98.0 (mixture of MS, βCD and TG) CUR retention (R%) in carbonated beverage: 53.8 (mixture of MS, βCD and TG); 96.6 (mixture IN, MD and G)	(Guo et al., 2020)
CH, TPP	T _{inlet} : 180°; T _{outlet} : 80°; Air pressure: 5 bar, FFR: 1.5 mL/min	25.0–225 nm	92.5 – 98.5	In-vitro release (without enzymes): - in SGF (2h): 0.97% - 3.97%; in SIF (24h): 1.21% - 4.21%; Pharmacokinetic studies (female Wistar rats): - maximum concentration (C _{max}) in plasma in 2h: 94 (conventional CUR suspensions), and 1034 (CUR loaded nanosuspension); - oral bioavailability: 2977 ng·h/mL (conventional CUR suspension) 33136 (CUR loaded suspension)	(Roopa et al., 2020)

PLGA, PVA, D-MA	Two-fluid nozzle: 0.7 mm, AFR: ~536 L/h, T _{inlet} : 65 °C, T _{outlet} < 40°C, Peristaltic pump 7%	0.5 – 5 µm	97.38	The photoinduced inactivation of bacteria by CURMPs studied; CURNPs and redispersed spray-dried CURMPs reduced the viability of <i>Staphylococcus saprophyticus</i> subsp. <i>bovis</i> and <i>Escherichia Coli</i> DH5 alpha.	(Preis et al., 2019)
MD/G of 26:0.6 (w/w)	Two-fluid nozzle: 0.7 mm; T _{inlet} : 124-190°C; AFR: 275 to 536 L/h FFR: 1.4 to 8.6 mL/min	NA	3.5 - 77	MC varied from 2.68 to 7.61 % and dried powder solubility ranged from 84.55% to 98.77%,	(Ferreira et al., 2019)
MD + GA mixtures	Two-fluid nozzle: 0.7 mm, T _{inlet} : 150, 175, 200°C, T _{outlet} : 90°C, Air pressure: 5 bar, FFR: 5 mL/min	1 – 20 µm	45.21 (MD, T ₁ = 200°C) - 73.67 (GA, T ₁ = 175°C)	Stability of CURMPs against light, oxygen and heat was higher than the nonencapsulated CUR; <i>In vitro</i> release showed the burst release in the first hour, followed by a prolonged release in one week	(Aniesrani Delfiya et al., 2015)
SPI SSPS, SSPS + MD mixtures	Two-fluid nozzle: 0.7 mm; T _{inlet} : 150°C; FFR: 3.5 mL/min, AFR: 667 L/min	55.1 – 68.8 nm	89.1 – 97.9	The encapsulated CUR exhibited a much higher oxygen radical absorbance capacity than free CUR (in DMSO); Both free/encapsulated CUR showed a remarkable cytotoxicity or anti-proliferation against human hepatal carcinoma HepG2 cells; Adding SSPS or MD to SPI decreased the bioaccessibility of encapsulated CUR.	(Chen et al., 2020)
SMC at 30% dry extract	Two-fluid nozzle; T _{inlet} : 161.6 – 165.3°C, T _{outlet} : 88.6-92.2°C, FFR: 35 – 52 mL/min	15 µm	NA	The skim milk powder without CUR exhibited lower wettability and solubility but a better dispersibility while the CUR-skin milk powder was not wettable and poorly dispersible; After 6 month storage at 25°C, the CUR loss was 28%.	(Neves et al., 2019)

SM, SO Tween 20	Two-fluid nozzle; Gas spray FFR: 630L/h; T _{inlet} : 135°C, T _{outlet} : 80°C, FFR: 6 mL/min	0.11 µm (D ₃₂)	17.7	Cytotoxicity of spray dried CUR on Caco-2 cells, showed a proliferative activity > 90%; Spray dried CUR uptake by Caco-2 cell monolayer revealed: - transepithelial electrical resistance: ≈140% - basolateral CUR concentration: 40.51 ng/mL	(Guri et al., 2018)
G, porous S	T _{inlet} : 190°C, AFR: 70 m ³ /h, FFR: 70 mL/min	NA	NA	The preservation of tofu, cooked pork and bread by CUR spray-dried MPs was studied	(Wang et al., 2012)
PVP Colloidal silicon dioxide	T _{inlet} : 110°C, T _{outlet} : 55°C	625-816 nm	NA	The flow parameters of CUR powder: - bulk density: 0.39-0.40 g/cm ³ ; tapped density: 0.52-0.56 g/cm ³ , - angle of repose: 9.6 – 14.8 - Carr's compressibility index: 25%; Hausner's ratio: 1.30 – 1.33	(Kaur et al., 2017)
MD, GA, MS, MD + GA, MD + GA + MS	Spray nozzle: 0.7 mm, T _{inlet} : 170°C, Air pressure: 6 kgf/cm ² , FFR: 3.9 mL/min, AFR: 536 L/min	NA	7.96 (MD:MS=50:50) to 45.23 (GA:MS=50:50)	MC: 0.84% (MD:MS=25:75) to 4.81% (MD:GA=75:25); Solubility: 85.35% (MD:GA:MS=66:17:17) to 99.25% (GA:MS=25:75)	(Cano-Higuera et al., 2015)
CAS, SUC, D-α-TPGS	T _{inlet} : 150°C, T _{outlet} : 90°C, Spraying pressure: 5.0–5.8 mbar, FFR: 5 mL/min, AFR: 320 L/h	NA	NA	The micellar-CUR powder was prepared by SD with drying yield from 60% (CAS:TPGS:SUC=42.5:1:25) to 92% (CAS:TPGS:SUC=42.5:1:0); Micellar- CUR solubility: 50% (CAS:TPGS:SUC=42.5:1:0) – 88% (CAS:TPGS:SUC=42.5:1:25)	(Wijani et al., 2020)
GEL, Colloidal silicon dioxide,	TSC: 7.5% (w/w), FFR: 5 mL/min, Atomisation air pressure: 4 kgf/cm ² , AFR: 1.5 m ³ /min, T _{outlet} : 40°C	550 µm	NA	CUR powder flow parameters: - angle of repose: 25°; Carr index: 9.53%; Hausner ratio: 1.10, - water activity: 0.315 Solubility of CUR: - free CUR: 0.75 µg/mL; spary dried dispersion CUR: 2700 µg/mL,	(Teixeira et al., 2016)

<p>- solubility of dispersions decreased 5,4% after 9 monthstorage; <i>In vitro</i> release of CUR: - 90% at pH =1.2 and 5.8 for 10 min; 73% at pH =7.4 for 10 min</p>	
<p>SC</p> <p>T_{inlet}: 105 °C, T_{outlet}: 68 °C</p> <p>Mean diameter after powder hydration: 168,7 nm</p>	<p>83.1</p> <p>Spray dried CURNPs had an AOXac almost 40 times higher than that of pristine CUR; Encapsulated CUR inhibited the tumor cells HCT-116 growth more than free CUR (Pan et al., 2013)</p>
<p>Mixture of: SMS, WPC, MD, GA</p> <p>Spray nozzle: 0.5 mm; T_{inlet}: 165, 180, 195°C; T_{outlet}: 80-85°C; FFR: 5 mL/min;</p>	<p>96.45(165°C) 96.96(180°C) 94.45(195°C)</p> <p>Particle size of CUR emulsions: 554.95 - 687.80 nm</p> <p>Increase of T_{inlet} resulted in increase of the powder wettability and decrease of the powder solubility, dispersibility, water activity and MC; CUR concentration in the microcapsules decreased with increase of T_{inlet}; Cumulative released CUR from CURNPs (%): - in SGF: 12.01; in SIF: 86.34 (Meena et al., 2021)</p>
<p>WPI</p> <p>The microfluidic jet spray dryer was used: T_{inlet}: 150°C, 110°C</p>	<p>95.0 (150°C), 97.5 (110°C)</p> <p>Particle size: 111.2µm (150°C) 108.4µm (110°C)</p> <p>The solubility of curcumin in 10% WPI aqueous solution was $f 124.9 \pm 4.8$ mg/mL; The freshly reconstituted 1% (w/v) aqueous solution of WPIcurcumin contained 10.9 ± 0.19 mg/mL curcumin for powder dried at 150°C and 11.3 ± 0.11 mg/mL for powder dried at 110° C; The AOXac of spray-dried WPI-CUR MPs was higher than that of WPI alone and increased with increasing curcumin concentration. (Liu et al., 2016)</p>

Nano spray drying					
Z, SC, P, CMC, GA, SA, CAR	Mesh size: 5.5 µm, T _{inlet} : 100°C, FFR: 120 L/min,	2 – 4 µm	70 – 80	CUR-loaded protein-polysaccharide complex NPs showed a slower release (45-70%) in simulated gastrointestinal fluids with enzymes: SGF (pH= 2; 2h); SIF (pH= 7; 4h); AOXac of CUR encapsulated in protein/polysaccharide NPs was better than free CUR SD of CUR-complex NPs substantially improved the redispersibility of powders in water	(Chang et al., 2017)
CH, Tween 20, Tween 80	4 µm spray mesh; T _{inlet} : 80, 100 or 120°C; AFR: 100, 120, 150 L/min;	285 – 360 nm	NA	Average Sp for samples with Tween 20, Tween 80, and samples without detergent were not statistically different; Both Sp and morphology were impacted most by the chitosan concentration, and AFR	(O'Toole et al., 2012)
Egg yolk LDL, P	T _{inlet} : 70, 100, or 120°C ; AFR: 130 L/min T _{outlet} : 50 – 60°C	54.6±0.7 nm (pH= 8; LDL/P 4:1) 914.4±93.2 nm (pH= 5; LDL/P:4:1)	100 (1, 2.5% CUR); 97.7 (5% CUR) 66.4 (7.5% CUR)	CURNPs were more stable in SGF at pH= 2 than in SGF at pH= 4 and SIF (pH= 7.5) At pH= 4 and 7.5, CUR release rate from NPs was slower than in gastric fluid at pH= 2.	(Zhou et al., 2016)
SA-sodium alginate; GA-gum arabique; IN-inuline; CH-chitosan; MCH-modified chitosan; WPC-whey protein isolate; G-gelatin; MD-maltodextrin; CAS-casein; SC-sodium caseinate; S-starch; MS-modified starch; SUC-sucrose; CAR-carrageenan; P-pectin; CMC-carboxymethyl cellulose; Z-zein; TPGS- tocopherol polyethylene glycol-1000 succinate; FFR-feed flow rate; AFR-air flow rate; SPL-soy protein isolate; SSPS- soy soluble polysaccharides; PVP- polyvinylpyrrolidone; PVA-polyvinylalcohol; SM- skim milk; SMC- skim milk concentrates; SMS-skimmed milk solid; SO-soy oil; GEL-Stearoyl macrogol-32 EP (Gelucire); PLGA-Poly(lactic-co-glycolic acid); LDL-low-density lipoprotein; TG- Tamarind gum; TPP- tripolyphosphate; β- Cyclodextrin (β-CD); D-MA-dextro manitol; SGF-simulated gastric fluid; SIF-simulated intestinal fluid; CUR-curcumin; CURNPs-curcumin nanoparticles; CURNPs-curcumin microparticles; AOXac-antioxidant activity; DMSO- dimethyl sulfoxide; Sp-particle size; MC-moisture content					

Chitosan is another biopolymer used to encapsulate CUR, due to its special functional and biological properties. Chitosan is a natural biopolymer, with amino-polysaccharide structure, produced by deacetylation of chitin from crustaceans and insects. In the chemical composition, chitosan contains free amino groups that protonate in an acidic environment, and allow it to interact with anionic polyelectrolytes, such as polysaccharides. It is a non-toxic, biodegradable, biocompatible, and bioadhesive compound with anti-inflammatory, antibacterial, antifungal, and hypoglycemic properties (Sánchez-Machado *et al.*, 2019). Studies have shown that particles with chitosan protect CUR against the aggression of physicochemical factors (light, oxygen, pH, moisture content (MC)) and improve cell absorption by helping to maintain its therapeutic effects (Akhtar *et al.*, 2012). Roopa et al. (2020) encapsulated CUR in chitosan NPs using tripolyphosphate as crosslinking agent, in the presence of Tween 80. The nanoparticulate dispersion was spray dried under $T_{inlet} 180^{\circ}\text{C}$; $T_{outlet} 80^{\circ}\text{C}$, air pressure of 5 bar, FFR 1.5 mL/min and drying process varied between 30-50 min. The EE of CUR in chitosan NPs ranged between 92.5 - 98.5%, and the Sp was 25 to 225nm.

Atomization

After preparation, FM is pumped into the atomizer, where it turns into very fine droplets, and sprayed into the drying chamber (DrC). Atomization is an important step in SD because it determines the size, shape, and morphology of particles. The size of droplets (Dd) formed by atomization depends on both factors related to the equipment and factors related to FM characteristics (Boelet *et al.*, 2020).

Atomization is performed differently in a cSD than a nSD (Arpagaus, 2018). In cSD, several types of atomizers are used including pressure atomizers, with a single-fluid high-pressure spray nozzle, pneumatic atomizers (two-fluid nozzle), and rotary atomizers. Pressure atomizers operate on the principle of the hydraulic nozzle which consists of pushing FM, under high pressure (250 - 10,000 Psi) through a pipe with a decreasing diameter (Figure 1 II.A). Thus, the fluid, at high pressure, comes out through a small hole (0.5 - 4 mm), with high velocity and disintegrates into small drops, when the pressure in DrC suddenly decreases. These atomizers produce large particles with average size (20 - 600 μm), less homogeneous, with higher density and allow for the use of vertical and narrow DrCs. The disadvantage of these atomizers is that they cannot be applied to high viscous FMs.

Two-fluid nozzle atomizers consist of two concentric cylinders through which FM (internal cylinder) and the atomizing gas (external cylinder) enter separately. The atomizing gas enters the nozzle under a pressure = 250 - 10,000 Psi. The two fluids can meet either inside the nozzle or outside the nozzle (Figure 1 II. B and C). Due to the very high speed of the atomizing gas, the frictional force between the gas and the liquid causes the liquid to break into fine drops sprayed into DrC in the form of a cloud. The atomization process is influenced by the properties of FM (concentration, TSC, viscosity, FFR) and the gas characteristics (pressure, speed, liquid/gas ratio, gas flow direction in relation to FM). These atomizers are also used in the lab spray-dryers, with small DrCs. The droplet size varies between 10 and 200 μm and can be controlled by adjusting the atomization parameters. The advantage of these

atomizers is that they allow the spraying of viscous and very viscous liquids, and the disadvantages consist in high cost, due to the high consumption of compressed gas, and the low density of MPs due to air occlusion. Rotary atomizers have a wheel or disc that rotates at 10,000 to 30,000 rpm. Under the centrifugal force, FM is pushed through some channels to the periphery of the wheel, and is removed through small holes forming a cloud of droplets. The advantages of these atomizers are the use of abrasive and very viscous FMs, the formation of particles with a uniform size between 10 – 200 μm , and the operation at low pressures with high efficiency.

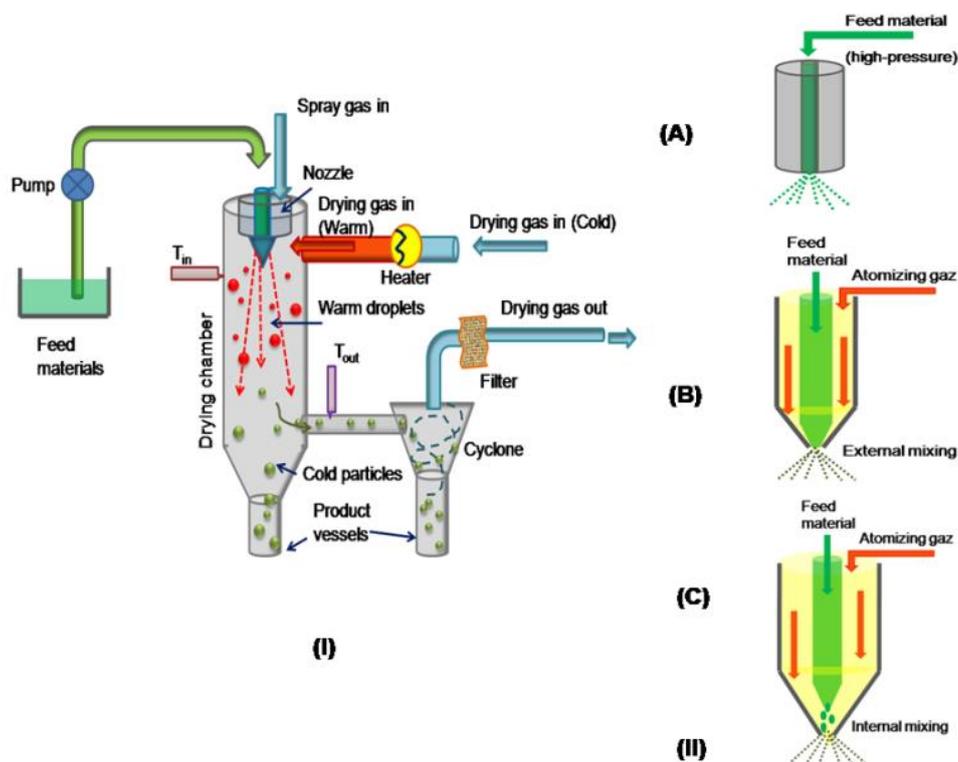


Figure 1. Conventional mini-spray dryer: schematic representation (I) and types of pressure atomizers (II): A: a single - fluid spray nozzle; B, C: two-fluid spray nozzle

In nSD, the classic atomizer is replaced by a spray head that works on the vibration principle of a very finely perforated metal membrane called mesh spray (Figure 2B). It contains over a thousand conical holes with a diameter of 50 μm towards the liquid supply side and with a diameter almost ten times smaller, on the side, through which the drops are released. The first generation of spray head used in nSD contained mesh spray with standardized holes and dimensions of 4.0, 5.5, and 7.0 μm (Arpagaus, 2018). Spray mesh is connected to a piezoelectric actuator that produces high frequency vibrations (80 - 140 kHz). By vibration, the spray mesh bends up and

down producing liquid rupture and the formation of droplets with an average diameter of 3 - 15 μm with a narrow droplet size distribution (SPAN = 1.11 to 1.32) (Arpagaus, 2019). The size of droplets depends on the diameter of the mesh spray holes and the liquid characteristics, e.g., concentration, TSC, viscosity, and surface tension (Nandiyanto and Okuyama, 2011). Vibration mesh atomization of liquids with a viscosity $> 5 - 10 \text{ mPa}\cdot\text{s}$ is very difficult because there is a risk of clogging some fine holes of the mesh spray (Arpagaus, 2019).

Drying of droplets and particle formation

After atomization, the fine droplets are sprayed into DrC where they come in contact with the drying gas and are converted into particles. Drying gas is usually atmospheric air, but in some cases, when FM is sensitive to oxidation, other inert gases, such as nitrogen, can be used. The design of DrC is done in relation to the type of atomizers, the direction of AFR, the properties of the particles and the collecting mechanism of the particles. Thus, DrC must have the appropriate size, shape, and height, to guarantee enough time necessary for the complete drying of particles, in order to avoid their sticking on the walls of DrC. In general, DrCs for cSD are vertical, with the cylindrical upper part, and the lower one in the form of an inverted cone through which MPs are collected. The hot gas can be introduced into DrC by co-current flow, when both the droplets spray and the gas enter at the top of DrC, flowing in the same direction; or by counter-current flow, when the droplets spray and AFR enter at opposite ends of DrC: the atomizer is positioned at the top and the gas is supplied from the bottom. In cSD, the gas enters DrC, with a temperature between 220 -350°C, and leaves DrC at 80-120°C (Assadpour & Jafari, 2019). The nSD has cylindrical DrC with different heights (0.32 or 0.77 m). The drying gas is heated to about 120°C by passing over an electrically heated porous metal foam. The porous metal surface ensures the laminar flow of the gas, thus avoiding the agglomeration of NPs or their deposition on the walls of DrC (Figure 2.). The nSD operates by co-current flow with AFR= 80 - 160 L/min (Arpagaus, 2019).

Two equilibrium transfer processes take place in DrC: the transfer of heat from the hot air to the liquid droplets due to the difference between air and droplet/particle temperature; and the mass transfer manifested by the release of the solvent from the droplet/particle, due to the difference between the vapor pressure of the liquid solvent and the partial pressure of the solvent in the gas phase (Santos *et al.*, 2017).

The drying time is a very important parameter in the preparation of particles by SD, which can be controlled both by modulating the technical characteristics of the atomizers and DrCs, and FM properties. The kinetics of particle formation by SD shows two main physical processes such as heat transfer from the drying gas to the liquid drops and mass transfer (diffusion and solvent evaporation) (Santos *et al.*, 2017)

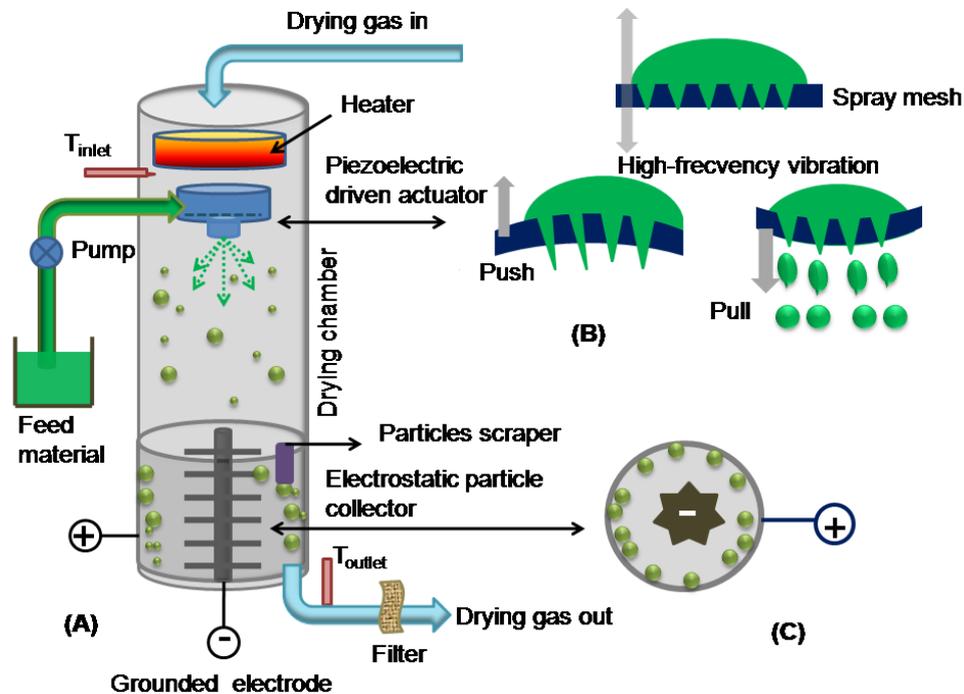


Figure 2. Nano-spray dryer: schematic representation of Büchi Nano Spray Dryer B-90(A); atomization by high frequency vibrations of spray mesh (B) and electrostatic particle collector (C). (Adapted from: Jafari *et al.*, 2021)

Particle collection

The collection of particles formed in DrC involves their separation from the drying gas. In cSD, heavier particles are recovered in the conical vessel at the bottom of DrC, while smaller and lighter particles are entrained by the gas stream outside DrC, where separation can be achieved using some collectors including the cyclone separator, the bag filter, or the electrostatic precipitator (Santos *et al.*, 2017). In nSD, the submicron particles are collected by electrostatic separation. At the bottom of DrC, there is an electrostatic particle collector, consisting of a metal cylinder, which acts as an anode and a star-shaped metal device, acting as a cathode, located inside the cylinder. A high voltage (15 kV) is applied between the two electrodes. The dry particles, which enter the electric field produced by the two electrodes, are negatively charged and are diverted to the inner wall of the cylinder, positively charged, where they are deposited in the form of a layer, and recovered using the particle scraper. Larger particles are deposited at the top of the inner wall of the cylinder because they have a larger surface area and consequently a higher electrostatic force. The efficiency of submicron particle separation in electrostatic particle collector is > 99% (Arpagaus, 2019).

Spray drying parameters and curcumin micro- and nanoparticle characteristics

The characteristics of particles are directly influenced by numerous parameters involved in SD, such as: parameters related to FMs (composition, surface tension, TSC and viscosity, solvent type, sample volume), parameters related to atomization (pressure, vibration frequency, spray rate intensity, spray cap type, spray mesh size), parameters related to drying gas (gas type, AFR, aspirator speed, drying gas humidity), T_{inlet} , DrC length, electric field, etc. (Arpagaus, 2018). The composition of FM and FFR directly affects the size and morphology of particles. The carrier material prepared from higher molecular weight substances involves higher viscosity solutions that lower FFR and lead to large particles. Temperature is an important parameter monitored at different stages of the drying process. Thus, FM is heated to 40 - 50°C in order to improve the homogenization process and decrease the viscosity corresponding to a good atomization. The T_{inlet} directly influences the drying rate, MC, density, and morphology of particles. Higher T_{inlet} leads to lower MC of the powder, and prevents particle agglomeration, and particles adhesion on the walls of DrC. The temperature of the drying gas is carefully controlled to avoid rapid evaporation of the solvent, glass transition in wall material, and the production of cracks on the surface of particles through which heat-sensitive bicomponent can be lost and degraded. When the drying gas temperature becomes over T_g of the wall material, the particle microstructure changes from glassy (amorphous) state to rubbery state. Therefore, in order to avoid the destruction of particles by stickiness, caking, and collapse, it is recommended to use wall material with T_g above the air-drying temperature (Piñón-Balderrama *et al.*, 2020). T_{outlet} is the highest temperature at which the drying process of particles ends. It is the result of the exchange of heat and mass during drying and corresponds to the air temperature measured at the exit of DrC, before collecting the powder. This output parameter is difficult to control because it depends on several input parameters, e.g. T_{inlet} , pressure and AFR, wall material composition, etc.

AFR expressed as the volume of hot gas entering DrC per unit time must be optimized to ensure, on the one hand, the time required for the complete drying of the droplets and particles, and, on the other hand, to improve the particle collection process. The residence time in DrC for the atomized droplets is an important parameter that influences the particle characteristics. This, in turn, it is influenced by the initial concentration and saturation concentration of the wall material, the evaporation rate, and the initial size of the droplets. In general, for FM with low viscosity, the residence time is 10-15s, when fine particles are obtained, with a minimum MC. For the high viscosity FM, the residence time is greater than 25 s and larger particles are obtained (Santos *et al.*, 2017). Numerous researchers have studied the influence of these parameters on important characteristics of free nCUR and CUR-loaded particles prepared by SD, such as: Sp and morphology, product yield, EE, loading rate, MC and flow properties, storage stability, and bioavailability (Table 2).

Particle size and morphology of spray dried curcumin

The shape, morphology, Sp and size distribution of particles are important characteristics that decisively influence the biological properties of CUR and the quality and safety of fortified foods. Thus, Sp affects their compatibility with the food matrix in which they are incorporated, the physical stability of the functionalized food, the bioaccessibility and bioavailability of encapsulated CUR (Dima *et al.*, 2020). The appearance of food functionalized with encapsulated CUR by SD method, including color, brightness, clarity, turbidity and sensory attributes (creaminess, smoothness, crunchy, etc.) largely depend on the size and morphology of particles (Gomes & Kurozawa, 2024). Moreover, the size and shape of particles obtained by SD influence the appearance, flowability, and dispersibility of the powders. The Sp of powders obtained by cSD may be classified into small (1–5 μm), medium (5–25 μm), and large size (10–60 μm). The Sp can be controlled by adjusting operational parameters. The properties of carrier material affecting the size and morphology of particles were discussed in the previous section.

Atomization is an important step that crucially affects Sp. For nozzle spray atomization, Sp is controlled by nozzle orifice size and the spray pressure of FM; for single-fluid spray nozzle and air flow for two-fluid nozzle, Sp varies inversely with the atomizing pressure. Thus, when working at a high atomizing pressure and with nozzles with small holes, smaller particles will be obtained. In most cases, these parameters are limited by the characteristics of DrC, such as its length, volume, and AFR. The optimal drying tower configuration of a nozzle atomization spray dryer corresponds to 3: 1 to 4: 1 height to width ratio (Jafari *et al.*, 2021) Moreover, when the drying gas flows in the same direction as FM, the drying tower must be higher to ensure the time required for complete drying.

The Sp obtained with nSD depends on the nature of the encapsulating material, the frequency of vibration, the size of the holes in the spray mesh, and the area of particle deposition in the electrostatic particle collector. As an example, O'Toole *et al.* (2012) prepared chitosan NPs loaded with CUR using Tween 20. The acidic chitosan and CUR solutions were atomized using a 4 μm spray mesh nozzle. The influence of drying parameters on the physicochemical characteristics of CUR-loaded NPs was investigated. For this purpose, the authors applied $T_{\text{inlet}} = 80, 100, \text{ or } 120\text{ }^{\circ}\text{C}$; AFR = 100, 120, or 150 L/min; chitosan = 0.025, 0.05, or 0.1 w/v%; Tween = 0, 0.025, or 0.05 w/v%. SD parameters were optimized using a Taguchi orthogonal array. The smallest Sp (285 nm) was produced using 0.25% chitosan, without Tween, at 120 $^{\circ}\text{C}$ T_{inlet} , and 120 L/min AFR, while the most spherical particles were produced using a 0.05% chitosan with Tween = 0.5 w/v%, at 120 $^{\circ}\text{C}$ T_{inlet} , and 100 L/min AFR. Using water-soluble polymers as encapsulating material, e.g., sodium alginate, GA, and modified chitosan (96.5% deacetylation degree), Lucas *et al.* (2020) prepared CUR-loaded MPs by cSD. The wall biopolymer solutions (1% w/v) were mixed with a 0.9% (w/v) solution of CUR in ethanol and atomized using a standard 0.5 mm diameter nozzle. The drying process was performed at $T_{\text{inlet}} = 150^{\circ}\text{C}$, FFR = 4 mL/min, AFR = 40 m^3/h and $T_{\text{outlet}} = 68\text{-}70^{\circ}\text{C}$. Product yield varied between 29-42%. Spherical MPs, with regular shape and smooth surface (modified chitosan) or rough

surface (alginate or GA) were obtained. CUR-loaded MPs prepared with modified chitosan were ten times larger than other CURMPs (Table 2).

Retention, encapsulation efficiency, and release of curcumin

The quality of powders obtained by SD is assessed by both retention (R) and encapsulation efficiency (EE), calculated with and without the amount of CUR retained on the particle surface. These two parameters are strongly influenced by both the size and morphology of particles, as well as the stability of CUR under SD conditions. Thus, particles with irregular surface, with many wrinkles, retain on their surface the CUR, and those with cracks can lose CUR that can degrade at drying temperature.

CUR is a heat-, pH- and light- sensitive compound. Therefore, the stability of CUR has been studied both in SD conditions and in the storage, processing, and digestion conditions of foods fortified with CUR. The rate of CUR retention is influenced by numerous factors, such as: condition of CUR and composition of carrier materials, Sp, morphology, and MC of powders. For example, to improve the solubility of CUR, Cano-Higueta *et al.* (2015) prepared CUR-loaded MPs by SD using MD, GA and MS alone or mixed in various ratios. GA powder had the lowest CUR retention (R% = 24.8) compared to other powders in which CUR retention values were greater than 60%, such as: 68.43% for GA: MS (25:75), 65.46% for MD: GA (25:75), and 61.98% for ternary mixture MD: GA: MS (17:17:66). All these powders showed EE between 30 and 33%, except for the formula MD: MS (50:50), which showed EE = 7.96% and R = 35.41%. The authors suggested that the low values of EE were due to wrinkles and cracks on the surface of particles or their deformation by wrinkling or even collapse.

Other researchers, such as Wang *et al.* (2009) reported encapsulating CUR, by SD, with EE = 98.4% using porous starch and gelatin as wall material, while Aniesrani Delfiya *et al.* (2015) encapsulated turmeric oleoresin, by the same method, in mixtures of MD and GA with different EE from 71.74 to 74.67%. Some studies showed that the state in which CUR was introduced into FM influenced both the EE and the release of CUR from particles. For example, Lucas *et al.* (2020) prepared CUR-loaded MPs using an aqueous solution of biopolymers (alginate, GA and modified chitosan) of the same concentration (1%) and a solution of CUR in ethyl alcohol (0.9%), while Bucurescu *et al.* (2018) prepared CUR-loaded MPs *via* an emulsion with CUR solubilized in coconut oil. CUR-loaded emulsions were stabilized with GA at different concentrations (10, 15, and 20% w/v). The EE of CUR was different depending on the nature and concentration of the biopolymer, but also on the condition of CUR in carrier materials. EE of CUR in alginate, GA and modified chitosan MPs was 97.0, 97.6 and 93.6% respectively, while MPs with GA and CUR in coconut oil had an EE between 75% and 85%.

The two teams mentioned above also studied the release of CUR from MPs to octanol. The kinetic parameters and transport mechanisms involved in the release process were evaluated using two mathematical models, Korsmeyer-Peppas model (eq.1) and the Weibull model (eq.2):

$$\frac{Q_t}{Q_\infty} = k \cdot t^n \quad (1)$$

$$\frac{M_t}{M_\infty} = 1 - e^{-\left(\frac{t-t_0}{\tau_d}\right)^\beta} \quad (2)$$

M_t and M_∞ in eq. (1) and (2) represent the cumulative amount of CUR released up to time t (min) and infinite time (min), respectively.

In equation (1), k is the Korsmeyer constant (dimension of time^{-n}), which incorporates structural and geometric characteristics of the released CUR from the sphere, film and cylinder delivery systems, and n is the transport exponent (dimensionless) that defines the release mechanism. In the Weibull equation (2), t_0 is the lag-time (min) of the releasing process (normally $t_0=0$), τ_d is the time (min) when 63.2% of M has been released and β is the „shape parameter of the curve” which characterizes the shape of the release curve (e.g. parabolic for $\beta < 1$; exponential for $\beta = 1$, or sigmoid for $\beta > 1$) and provides insight into the release mechanism. Diffusion parameter values, n , reported by the authors, suggested that the release of CUR from MPs prepared with alginate and modified chitosan was a controlled diffusion process ($n < 0.43$), while the release of CUR from MPs prepared with GA took place by anomalous transport (case II transport), controlled by both diffusion and swelling process ($0.43 < n < 0.85$) (Corsaro *et al.*, 2021).

O’Toole *et al.* (2012) prepared chitosan NPs loaded with CUR using a nSD with a 4 μm spray mesh. They studied the release of CUR in two release media: 1% acetic acid and 1% PBS buffer, fortified with 1% ascorbic acid and 0.1% butylated hydroxytoluene (BHT), in 1-octanol as preservatives. The release of CUR from chitosan NPs, in the two solvents, was rapid in the first 5 min of the experiment when about 40% of CUR was released, and after 2 h, the release was complete.

Other authors studied the release of CUR in simulated fluids of the gastrointestinal tract (GIT) and evaluated the oral bioavailability of CUR encapsulated by SD. For instance, Roopa *et al.* (2020) studied the release kinetics and oral bioavailability of conventional CUR suspension and CUR encapsulated in chitosan NPs obtained by SD. Accordingly, in simulated gastric fluid (SGF), the release rate was between 0.97% and 3.97%, after 2 h digestion, while in simulated intestinal fluid (SIF), the release rate varied between 1.21% and 4.21%. In SIF, after 2 h digestion, the concentration of CUR decreased significantly due to the alkaline degradation of CUR. Conventional CUR suspension showed a burst release (95%) after 2 h in SGF. For the *in vivo* study of the CUR bioavailability, six female Wistar rats were used and pharmacokinetic parameters with different values for conventional CUR suspensions and CUR-loaded NPs were reported. The maximum concentration (C_{max}) of plasma CUR, 2 h after administration of CUR-loaded NPs was 10-fold higher than for conventional CUR suspension, while the oral bioavailability of encapsulated CUR was 11-fold higher than for the unencapsulated CUR.

An interesting study was published by Zhou *et al.* (2016) on the stability and release of CUR encapsulated by pH-driven process and nSD. Firstly, the authors prepared CUR nanogels from pectin and egg yolk low-density lipoprotein by adjusting the pH

and heating processes. The formed nanogels were then converted into a fine powder loaded with CUR, by applying drying conditions of $T_{inlet} = 70, 100, \text{ or } 120^{\circ}\text{C}$, $AFR = 130 \text{ L/min}$, and $T_{outlet} = 50 - 60^{\circ}\text{C}$. To obtain CURNPs with improved characteristics, the authors optimized the operational parameters, the pectin: protein mixing ratio and the pH value, depending on the pI of the components. CURNPs with a size of 54.6 nm to 914.4 nm were obtained. The authors studied the behavior of CUR-loaded NPs in SGF with pepsin and SIF with pancreatin. In SGF, at pH 2, the NP size increased by almost 73 nm, while at pH 4, the increase in NP size was only 16 nm. This is because at pH 2 pectin is strongly protonated, and the electrokinetic potential decreases, causing the aggregation of NPs. When maintaining CURNPs in SIF (pH 7.5), for 4h, the size of NPs did not change significantly, due to the stabilizing effect of salts and surfactants in SIF. The release of CUR from NPs was studied under the same conditions as the stability of NPs. The results showed that at pH 2, the release of CUR was faster than at pH 4. Compared to free CUR, whose diffusion was rapid, reaching a maximum of 70% after 2 h, the cumulative rate of CUR released from NPs in the first 2 h was 50% at pH 2 and 25% at pH 4, respectively. Under SIF (pH 7.5), the kinetic profile of free CUR corresponded to a first-order mechanism, with a cumulative release rate of 65% after 3h digestion, while the rate of CUR release from NPs was only 20% in 3h.

Rehydration of spray dried curcumin powders

Rehydration is an important property of food powders in terms of both the quality of reconstituted foods, such as reconstituted milk, infant formula, cocoa, and high protein drinks, and the bioefficiency of CUR encapsulated and incorporated into food matrices. Several processes are involved in rehydration of powders, e.g., wetting, immersion, swelling, dispersion, and solubilization of particles. Wetting is the relationship that is established between the surface of particles and water molecules. Some particles get wet more easily and others harder, depending on the composition and morphology of the particle surface. Thus, spherical particles with a smooth surface, prepared from hydrophobic materials, are more difficult to wet than particles with an irregular shape, with a rough surface, prepared from hydrophilic materials. The hydrophobicity of particles obtained by SD method can be manipulated by adjusting the operational parameters and FM composition (Dima *et al.*, 2016). It was found that the powders prepared at high SD temperature and with the high TSC in wall material were easily wettable. The presence of fats on the surface of particles and the degradation of proteins during SD determines the decrease of wettability in powders (Murrieta-Pazos *et al.*, 2012). Moreover, the wetting of powders depends on the absolute density, S_p , water temperature, and liquid agitation. Higher density powders tend to sink into water faster, being heavier than water, and larger particles get wet more easily due to the larger spaces between particles that allow water to penetrate more easily through the capillarity phenomenon (Fitzpatrick *et al.*, 2017). Agitation of the wetting liquid is an important factor that influences the wettability of hydrophobic particles, preventing their agglomeration and the formation of difficult disperse clumps. The liquid temperature influences the wettability by changing the surface tension, the hydrophobicity of the

wall material and the contact angle. Moreover, wettability can be altered by storage and processing of powders due to phase transformations of substances in the wall material, which cause changes in particle hydrophobicity and contact angle (Fitzpatrick *et al.*, 2016).

After wetting, the powders are immersed in the liquid, and the particles disperse and dissolve. Formulation of nCUR and encapsulation of CUR in particles by SD are effective ways to improve the solubility of CUR and incorporate it into various food matrices, especially in beverages. Several researchers have studied the dispersibility and solubility of nCUR prepared by SD, using different carriers (Table 2). For example, Neves *et al.* (2019) investigated the rehydration conditions of CUR-loaded NPs obtained by SD, using skim milk as wall material. According to their results, both raw milk powder and milk powder loaded with CUR showed low dispersibility (~ 30%) and high solubility (~ 95%). In contrast, skim milk powder loaded with CUR was poorly wettable (wettability time was over 45 min) compared to skim milk powder without CUR, for which the wetting time varied between 30 and 35 min. The authors suggested that the low wettability of CUR-loaded powders is due to the interaction of CUR on the surface of particles with casein, which changes the surface hydrophobicity and affects the interaction of powder with water.

Other authors encapsulated CUR by SD in SPI (Chen *et al.*, 2020a). By dispersing and dissolving SPI NPs loaded with CUR in water, transparent and yellow reconstituted dispersions were obtained. The dissolution capacity of CUR-loaded NPs varied depending on the composition of the encapsulating material; e.g., the addition of SSPS and /or MD, alone or in a combination, improved the water-dissolving capacity. The dissolution potential of NPs with MD alone was better than those with SSPS, because MD is more soluble in water. For the study of dissolution, mini-tablets were prepared by pressing the powder with CUR. These were added to a PBS at pH 6.8, at 37°C, and stirred with a paddle at 100 rpm. After the 30 min dissolution, different values of CUR solubility were obtained depending on the composition of the encapsulating material. When adding sucrose to casein, the solubility of CUR increased from 50%, for sucrose-free NPs to 89% for sucrose NPs, possibly due to the contribution of sucrose in maintaining the amorphous state of CUR, and also the formation of molecular nanostructured-level of CUR/sucrose composite during SD. The MC of powders can influence the solubilization capacity of CUR-loaded particles too, as revealed by Cano-Higueta *et al.* (2015) who encapsulated oleoresin turmeric, by SD using GA, MS, and MD alone and in combinations, obtaining powders with different MC. Powders prepared with GA:MS (50:50) showed a MC = 2.14% and a solubility = 95.63%, while powders prepared with GA:MS (25:75) had 1.58% MC and solubility = 99.25 %.

Flowability of spray dried curcumin powders

Flowability is an important feature, which decisively influences different powder handling operations, e.g., transportation, storage, mixing, formulation, compression, and packaging (Murrieta-Pazos *et al.*, 2012). A powder that does not flow well is very difficult to transfer from one place to another by gravity. The most common method of flowability measuring consists of freely flowing the powder through a

funnel and measuring the angle that the powder pile slope makes with the horizontal (angle of repose). The smaller the angle, the better the flowability (Jafari *et al.*, 2021). Flowability is dependent on particle shape, Sp and size distribution, surface chemical composition, density, etc. Particles with spherical geometry and high density exhibit the best flowability. The presence of grease on the particles surface makes it difficult for the powders to flow. To evaluate the density of powders, the density of particles (absolute density), the poured bulk density (D_B), and the tapped bulk density (D_T) are measured. Absolute density represents the weight of a given particle volume of powder, and bulk density (D_B) represents the weight of a given total volume of powder. Absolute density is influenced by the SD operational factors, such as FM, drying temperature, atomization process, Sp, which favor the inclusion of air in the particles and reduce their density. A high TSC in the wall material leads to an increase in the absolute density of powder. High density particles get wet more easily because they penetrate the water surface faster.

The powder flowability is evaluated using empirical parameters such Carr's index (CI) and Hausner Ratio (HR), which are calculated with the values of the bulk density (D_B) and the tappet bulk density (D_T) of powder (eqs 3 and 4):

$$CI = \frac{D_T - D_B}{D_T} \times 100 \quad (3)$$

$$HR = \frac{D_T}{D_B} \quad (4)$$

The *CI* is the measure of the cohesiveness between particles and explains the ability of particles to agglomerate, and the *HR* expresses the degree of friction between particles. The lower the *CI* and *HR* values, the better flowability (Chang *et al.*, 2017). *HR* and *CI* are strongly influenced by MC, Sp and shape, chemical composition of the particle surface, etc. A high MC increases the cohesiveness of particles and decreases D_B of powders, while the presence of anti-caking agents (calcium stearate, tricalcium phosphate, and silicone oxide) reduces interparticle forces and cohesiveness, and increases D_B of powders (Dima *et al.*, 2016).

Some researchers who prepared CUR-loaded particles by SD, studied the flow properties of powders and their modification during storage. For example, Kaur *et al.* (2017) prepared CUR-nanosuspensions by antisolvent precipitation method, followed by SD. The authors used ethanol as a solvent for CUR and water as an antisolvent for nucleation and precipitation. CUR precipitation occurred in the presence of sodium lauryl sulfate and PVP K-60, as solubilizers. After SD dispersions, CUR-loaded NPs with size of 500-700 nm and electrokinetic potential between -24 and -31.6 mV were obtained. In terms of flow properties of CUR powders, the authors reported *CI*: 25%, and *HR*: 1.30 and showed that the powders were passable flow powders. CUR-Gelucire® solid dispersions prepared via SD by Teixeira *et al.* (2016) showed also excellent solubility in aqueous HCl solution, 3600 times higher than free CUR, good stability during 9 months of storage, an increase in pharmacokinetic parameters compared to unencapsulated CUR and better anti-inflammatory activity. The particles of CUR-solid dispersions were irregularly spherical in shape, with a porous surface and an average D50 diameter of 550 μm .

The powders had an excellent flow, according to the values of flow parameters: angle of repose = 25°, CI= 9.53% and HR= 1.10.

Application of spray-dried curcumin powders in food

CUR and turmeric oleoresins/essential oils have been accepted since 2016 by the US Food and Drug Administration (FDA) as Generally Recognized as Safe (GRAS) materials. Over 60% of the global CUR market is used in the pharmaceutical and cosmetics industries, and only a quarter is used in the food industry (Sharifi-Rad *et al.*, 2020) mainly as a coloring agent and antioxidant, but it also contributes to increasing the shelf life of food due to its antibacterial properties. To ensure the necessary nutritional intake, CUR content varies between 5 - 500 mg/kg depending on the type of food prepared with CUR. The most common foods enriched with CUR are beverages (e.g. golden milk), and dairy products, along with confectionery, cereals, bakery products, and even fish, meat and meat products. The use of CUR in food is restricted by several obstacles: (i) consumers' preference for CUR-enriched foods is selective due to spicy taste and strong specific odor when using higher doses of CUR; (ii) the poor water solubility of CUR (11 ng/mL - 10 µg/mL) which makes it difficult to include CUR in the food matrix; (iii) the relatively high hydrophobicity of CUR and its low chemical stability in GIT fluids, which leads to a low CUR bioavailability. To eliminate these shortcomings, food and drug manufacturers have developed several strategies to improve the solubility, chemical stability, and bioavailability of CUR, the most effective one is encapsulations of CUR (Araiza-Calahorra *et al.*, 2018; Kanwal *et al.*, 2023; Rafiee *et al.*, 2019). In this section, applications of spray-dried CUR as coloring, antioxidant, and antibacterial agent in foods will be discussed.

Spray-dried curcumin as food coloring

Dyes are additives that help improve the sensory attributes of foods and drugs. Lately, more and more natural dyes are being used in food coloring, due to the low risk of toxicity and the increase in consumers' preferences for safe and healthy food. The main disadvantage of natural dyes is their low stability during food processing and storage. Due to oxidative or enzymatic degradation, many natural dyes change color over time, which leads to deterioration of food quality. CUR is one of the most widely used natural dyes in the food and pharmaceutical industries, accepted by FDA in 2016 under the codes E100 and 21CFR73600 (Code of Federal Regulations Title 21. Part 182). European Food Safety Authority (EFSA) and Joint FAO/WHO Committee of Experts on Food Additives (JECFA) periodically reviewed the amount of CUR accepted in food both as a colorant and as a spice. CUR-acceptable daily intake (ADI) was from 0-1mg/kg body weight (bw)/day, based on the no-observed-effect levels (NOEL) of 220 mg/kg bw/day to 0-3 mg/kg bw/day, for a NOEL value of 250-320 mg/kg bw/day (Commission of the European Communities, 1975; EFSA 2014; JECFA, 2004).

Research on the mechanism of food coloring has shown that the color of CUR is dependent on pH and the degree of protonation of its hydroxyl groups. In the pH

range of 2-7, found in most foods, CUR is golden yellow due to the complete protonation of hydroxyl groups. At these pH values, CUR is uncharged and has low water solubility. As pH increases from 7 to 8.5, the enol hydroxyl groups deprotonate, and CUR becomes negatively charged, and is soluble in water, developing a brownish-orange color; while at higher pH values the color of CUR becomes reddish (Zheng *et al.*, 2018; Zheng *et al.*, 2019). Some researchers evaluated the color of particles loaded with CUR obtained by SD in the solid state and other investigated the color of dispersed CUR particles in various simulated liquid food systems or in food matrices. The color characteristics of CUR encapsulated by SD are expressed in most studies in the CIELAB or CIE 1976 $L^*a^*b^*$ color space (Segura *et al.*, 2017). To determine the trichromatic parameters of CUR particles, specific colorimeters were used that measured the light reflected by the solid powders under standard optical conditions or spectrophotometers that measured the absorbance or transmittance of the colored liquid systems. Accordingly, the color of spray-dried CUR in powders or simulated liquids or food matrices varies depending on several factors, e.g., chemical composition of wall material, Sp and size distribution, retention and EE, release conditions (pH, ionic strength, enzymes), the nature of the food matrix (solid, liquid), the food processing, and storage conditions (time, temperature).

For example, Guo *et al.* (2020) evaluated the color of CURMPs prepared by SD using various encapsulating materials including corn MS, inulin, MD, pectin, β -cyclodextrin, and gelatin. CUR powders showed high yellowness ($b^* = 75.12 - 91.48$); the highest b^* was for CURMPs with lower EE and higher retention. The authors suggested that these results were due to the high CUR content remaining on the particles surface which contributed more to the yellowness than the encapsulated CUR. The spray-dried CURMPs were also used to fortify a carbonated beverage, e.g. Sprite, in which the pH varied between 3 and 4. After two days of storage, the liquid samples remained clear, yellow, and MPs showed a retention of approximately 90%, with higher values for MPs prepared with inulin mixed with MD and gelatin. Other authors studied the change in the color of CUR encapsulated by SD during storage. For instance, Neves *et al.* (2019) evaluated the stability of CUR encapsulated in skim milk during storage of dry powder at 10, 25 and 50°C for two months in hermetically sealed plastic bags, at 0.43 water activity. The authors correlated the color parameters with CUR content of the samples. The yellow color of CUR changes over time to brownish/reddish color, corresponding to the degradation products of CUR. During storage, L^* of CUR-powder decreased over time at all storage temperatures. At 10 and 20°C, the decrease of L^* was insignificant after 60 days, but a dramatic decrease of L^* was observed at 50°C when it decreased from 93-95 at zero time, to 20 -25, after 60 days, which means that the darkening phenomenon was more intense at this temperature. Moreover, the color saturation (C^*) remained unchanged at 10 and 20°C; instead at 50°C there was an increase in C^* for CUR free powder and a slight decrease for powder with CUR. The authors suggested that the change in color parameters of skim milk powders with or without CUR is mainly due to the Maillard reaction that takes place between the amino groups of proteins and the reducing sugar in milk resulting in brown compounds.

The yellow color of CUR powders tends to be maintained even after 60 days of storage at 10, 20 and 50°C, which also corresponds to the CUR content remaining in the powders at the three temperatures, such as 83, 72, and 58% respectively. At the same time, analyzing the relationship between chroma (C^*) and CUR content, the authors revealed that the presence of a certain amount of CUR in milk powder could limit the Maillard reaction. It was also suggested that milk powder protects CUR from oxidation. It was concluded that the presence of CUR in milk powder does not alter the physicochemical characteristics of the powder, such as Sp and size distribution, MC, water activity, solubility, and dispersibility, except for color which remains stable during storage only in the temperature range of 10 to 20°C.

Spray-dried curcumin as a food antioxidant

Many diseases, such as cardiovascular and neurodegenerative diseases, rheumatoid arthritis, asthma, cataracts, and cancer, are caused by the body's inability to defend itself against free radicals, e.g., reactive oxygen species (ROS) and reactive nitrogen species (RNS), which causes cell destruction by oxidizing components including lipids, proteins, cholesterol, and DNA (Chandimali *et al.*, 2025). To reduce oxidative stress and prevent the occurrence of chronic diseases, the human body has a defense system composed of various enzymes with AOXac, such as catalase, superoxidismutase, glutathione peroxidase, glutathione S-transferase, etc. However, in order to win the war with free radicals, the intake of exogenous antioxidants is also needed, which are introduced into the body by consuming foods rich in vitamin C, vitamin E, glutathione, lipoic acid, polyphenols, and carotenoids. CUR is a polyphenol whose AOXac is due to its chemical structure, mainly phenolic groups, and beta-diketone moiety, which allow for the donation of hydrogen atoms or electrons capable of neutralizing free radicals (Barclay *et al.*, 2000). The AOXac of CUR is manifested either directly by scavenging free radicals or quenching singlet oxygen, or indirectly by improving the activity of antioxidant enzymes or by inhibiting the pro oxidant activity of enzymes e.g., cyclooxygenase and lipoxygenase, xanthine oxidase and xanthine hydrogenase, or by chelating metal ions (Fe^{2+} , Fe^{3+} , Cu^{2+}) (Kanwal *et al.*, 2023; Rao, 2007; Zheng & McClements, 2020).

Due to its poor water solubility and chemical instability, pure CUR has low AOXac, which limits its use in food. Many studies published in recent decades have shown an increase in AOXac of CUR encapsulated in various carriers (Joung *et al.*, 2016; Changet *et al.*, 2017; Almeida *et al.*, 2018). Some authors have prepared CUR-loaded particles by SD and evaluated AOXac by various methods, such as: DPPH scavenging; ABTS (3-ethylbenzothiazoline-6-sulfonic acid) assay, Cupric Reducing Antioxidant Capacity assay (CUPRAC), and Oxygen radical absorbance capacity assay (ORAC) (Piñón-Balderrama *et al.*, 2020). AOXac was expressed either as a rate of free radical scavenging by CUR (AA%) or in terms of inhibitory concentration 50% (IC_{50}) of DPPH or exhibitory concentration 50% (EC_{50}) of ferric reducing antioxidant power (FRAP), DPPH, β -carotene, and TBARS. Sometimes, AOXac has been expressed in equivalents of standard antioxidant substance, e.g. Trolox Equivalent Antioxidant Capacity (TEAC) and Gallic Acid Equivalent (GAE). Trolox and gallic acid are synthetic compounds with high AOXac.

In order to increase the solubility and improve the biological activities of CUR, Pan *et al.* (2013) encapsulated CUR in micellar casein functionalized by SD. Water solubility of CUR in micellar casein was forty times higher than pure CUR. The AOXac of free and encapsulated CUR was determined by ABTS assay, using Trolox as a standard antioxidant. The AOXac of encapsulated CUR was 8.87 mM TEAC, almost 40 times higher than that of pure CUR (0.240 mM TEAC). These values were explained by the high solubility of encapsulated CUR which favored its reaction with free radicals derived from 2,2'-Azinobis (ABTS). Other authors reported opposite results (Khanji *et al.*, 2018). They measured AOXac of CUR in micellar casein powders by ABTS and FRAP assays and showed that AOXac of encapsulated CUR was 88% of active CUR and decreased to 82-84% after 60 days of storage at 40°C. The authors explained lower AOXac of encapsulated CUR due to the trapping of CUR inside casein micelles. Martin *et al.* (2013) encapsulated CUR by SD using a mixture of polyvinylpyrrolidone (PVP) and colloidal silicone dioxide. The authors optimized SD parameters and obtained CUR-loaded powders with a water solubility one hundred times higher than that of free CUR. The AOXac of encapsulated CUR was assessed by DPPH assay. The IC₅₀ ranged from 530 to 860.3 µg/mL depending on the ratio of PVP to CUR and the colloidal silicon dioxide content used to avoid the sticking FM and improve powders flowability. Other authors measured AOXac of free CUR dissolved in DMS or methanol and CUR encapsulated by SD method in SPI-NPs using ORAC and DPPH assays (Chen *et al.*, 2020). The free CUR in DMSO had an ORAC value = 5.6 µmol Trolox equivalent/µmol CUR, and the encapsulated CUR had 10.7 µmol Trolox equivalent/µmol CUR, higher even than the sum of the ORAC value of free CUR and SPI. In contrast, DPPH scavenging rate (IC₅₀) of free CUR in methanol and encapsulated CUR had close values, 40.4 and 36.8 µg/mL, respectively.

Spray-dried curcumin as an antibacterial agent

CUR helps to prolong the shelf life and increase food safety due to its ability to inhibit the growth of microorganisms. Numerous *in vitro* and *in vivo* studies have highlighted the antimicrobial potential of CUR against a wide range of bacteria, viruses, fungi, and parasites (Adamczak *et al.*, 2020). Researchers highlighted various mechanisms by which CUR manifests its antibacterial activity. Some researchers showed that the antibacterial effect of CUR is due to inhibiting the cytokinesis process in which the FtsZ protein is involved (Rai *et al.*, 2008). Accordingly, the chemical structure of CUR molecule plays an important role in the inhibition mechanism of bacterial cell division of *Bacillus subtilis* 168. It was suggested that oxygen atoms of phenolic hydroxyl groups (free or methoxylated) and carbonyl groups in the structure of CUR may form hydrogen bonds and hydrophobic interactions with the FtsZ protein, and prevent the protein self-assembling into a microtubule, called Z- ring, thus inhibiting the proliferation and growth of bacterial cells. Other authors revealed antibiofilm activity of CUR through the inhibition of bacterial quorum sensing systems (Vaughn *et al.*, 2017) and others have associated the bactericidal action of CUR with the damaging of bacterial membranes (Tyagi *et al.*, 2015).

As with other natural extracts, the antibacterial effect of CUR was evaluated mainly by two *in vitro* methods, such as the diffusion and the dilution methods (Jorgensen & Ferraro, 2009; CLSI 2015). The diffusion method performs a qualitative evaluation of antimicrobial activity, while the dilution method offers the possibility to quantitatively evaluate the antimicrobial activity expressed by the minimum inhibitory concentration (MIC) (Hossain, 2024).

The MIC of CUR reported by different authors varied depending on the condition of CUR (pure CUR, oleoresin, free or encapsulated CUR), the solvent used to extract CUR and of course the type of bacteria. As an example, the antibacterial activity of CUR was higher against Gram-positive bacteria than Gram-negative ones whose cells are protected by lipopolysaccharides in the cell membrane (Shlar *et al.*, 2017). The methanol extract of turmeric revealed MIC = 16 and 128 $\mu\text{g/mL}$ against *B. subtilis* and *Staphylococcus aureus*, respectively, while ethanol extract of turmeric exhibited a significant antibacterial activity against other pathogenic bacteria, such as *E. coli*, *L. monocytogenes*, *E. faecalis*, *L. innocua*, etc., which play an important role in food safety (Thomas *et al.*, 2024; Wu *et al.*, 2024). Like other biological properties, the antimicrobial activity of CUR is severely limited by its low solubility and chemical instability. Among the strategies to improve these characteristics, encapsulation of CUR has been widely exploited lately (Vaughn *et al.*, 2017; Silva *et al.*, 2018b; Kanwal *et al.*, 2023). However, there are few works in the literature studying the antimicrobial activity of free and encapsulated CUR by SD with applications in food fortification.

Amrouche *et al.* (2010) encapsulated CUR in MD by SD and investigated the synergy of CUR and subtilisin. The authors showed that encapsulated CUR had a higher antibacterial activity than its free form against *B. subtilis*, and subtilisin presented synergy with encapsulated CUR against *Listeria monocytogenes* Scott A. Other authors added an aqueous CUR extract (0.3%) to the cheese and found growth inhibition of various bacteria, such as *Salmonella typhimurium*, *Pseudomonas aeruginosa*, and *Escherichia Coli* 0157: H7. A decrease of *S. aureus*, *Bacillus cereus*, and *L. monocytogenes* contamination after 14 days of cold storage was also observed (Hosny *et al.*, 2011). Wang *et al.* (2012) evaluated the level of contamination of some foods treated with CUR encapsulated by SD, using gelatin and porous starch as wall material. Foods such as tofu, bread, and cooked pork were cut into small pieces and immersed for 5 min in a dispersion containing CURMPs at various concentrations. After drying and incubation at 37°C for 24, 48 and 72h, the mildew levels of treated food were determined. To evaluate the influence of temperature on antibacterial effect of encapsulated CUR, the authors first boiled the CUR dispersions for 20 min and then immersed the food samples. After 72 h of storage of treated food, the antimicrobial activity of encapsulated CUR was better than the one of free CUR, at concentrations of approximately 0.030%. Also, upon heating, the antimicrobial effect of encapsulated CUR was better than the one of free CUR whose chemical stability was affected by temperature.

Recent studies have investigated the phototoxic activity of CUR against Gram-positive/negative bacteria. The phototoxicity of CUR is due to keto-enol

isomerization, under the action of white light radiation (300 - 500 nm). The enol-tautomer corresponds to the excited state of CUR molecule and is able to transfer from its energy to oxygen molecules which it transforms into highly reactive oxygen species. These species can react with cell membrane components (unsaturated lipids, amino acids in proteins) causing cell death. The antibacterial activities of CUR as a photosensitizer has applications mainly in antimicrobial photodynamic therapy and less in the food industry. Chen *et al.* (2020b) prepared edible films based on CUR-functionalized polyvinyl acetate and studied antimicrobial photodynamic activity upon white light irradiation at 20, 40, and 60 mW/cm² intensity, against *S. aureus* (Gram-positive) and *S. typhimurium* (Gram-negative). Bacteria killing efficiency was better in the irradiated samples than in those kept in the dark and increased at higher CUR levels and irradiation intensity. An interesting study by Preis *et al.* (2019) revealed the photoinduced inactivation of bacteria based on CURMPs obtained by SD. The authors first incorporated curcumin into poly (lactic-co-glycolic acid) (PLGA) NPs. These NPs were then coated with mannitol by SD and led to easily redispersible CURMPs. In order to test the antibacterial activity of encapsulated CUR, the dilution method was used, and the dispersions containing CURMPs were irradiated at 457 nm, for 10 min. The CURNPs and redispersed spray-dried CURMP significantly reduced the viability of both Gram-positive (*Staphylococcus saprophyticus subsp. bovis*) and Gram-negative bacteria (*E. coli* DH5 alpha). However, the antibacterial effect, after photoactivation of the encapsulated CUR, was better against Gram-positive bacteria than Gram-negative ones.

Conclusions

Despite its limitations in terms of strong smell, poor solubility, and low bioavailability, CUR still remains the “golden spice” for Asian cuisine, and a natural compound with important contributions to food functionalization or the treatment of diseases. Over the last fifty years, research on CUR has intensified. Some researchers have studied CUR delivery systems, while others have investigated the therapeutic activity of CUR, highlighting the mechanisms involved in cellular uptake, metabolism, and bioavailability. Thus, CUR has been administered in various formulations, such as NCs, nanoemulsions, liposomes, NPs, tablets, capsules, etc. The colloidal micro/nanosystems of CUR delivery ensure the protection of CUR against light, oxygen, and pH variations, have a large interphase surface contributing to higher solubility, release rate and improved AOXac, antimicrobial and food coloring properties.

SD is one of the techniques used extensively in obtaining particles loaded with CUR. This technique has been used, on the one hand, to formulate uncovered CURNCs, and, on the other hand, to encapsulate free CUR or to incorporate colloidal systems loaded with CUR in various encapsulating materials. CUR encapsulation by SD was done with the highest yield and EE. For nanoencapsulation of CUR, an innovative SD has recently been developed, namely nSD, whose operations and advantages are briefly presented in this paper. The characteristics of particles obtained by cSD and

nSD, such as size, morphology, electric charge, hydrophobicity, as well as the properties of powders (redispersibility, solubility, density, flowability, etc.) can be controlled by the operational parameters of SD, and FM characteristics, depending on the requirements of the final application. Spray-dried nCUR has been used more in the pharmaceutical industry than in the food industry.

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