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# SPRAY CONGEALING: A VERSATILE TECHNIQUE FOR PARTICLE PRODUCTION

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**ABSTRACT**

Spray congealing is also known as "spray cooling/ spray chilling". This method is used to obtain microparticles (MPs). The pharmaceutical industry continues to grapple with a significant concern, characterized by the inconsistent and often low oral bioavailability of poorly water-soluble drugs. Spray congealing is a technology for the production of solid dispersion to enhance the bioavailability of poorly soluble drugs by using low - melting hydrophilic excipients. Spray congealing is a simple technique, less time and energy consuming. It consists in a liquid atomization through a hot gas current to directly obtain a fine and dry powder. The aim of this review is to illustrate the potential of spray congealing in the preparation of microparticles aiming to enhance the oral bioavailability of poorly water-soluble drug. Spray congealing has been recently proposed as an emerging technology for the manufacturing of solid dispersion in the form of microparticles. Spray congealing is a dynamic and versatile process with applications spanning across various industries. This review article provides an in-depth exploration of the fundamentals, applications, advantages, challenges, and emerging trends in the field of spray congealing. Furthermore, an evaluation of the current state of industrial technology applications in the pharmaceutical sector is examined. The final part points out benefits, limitation and future perspective of this technology in drug delivery.

**KEYWORDS:** microencapsulation, atomization, solubility enhancement, spray cooling, dissolution rate.

## INTRODUCTION

Spray congealing it is also known as spray cooling. It is a solvent free process that helps to transforms a melt into well-defined spherical particles. In recent years, spray congealing technology has been used for controlled release formulation.[1] This technique is also use for taste masking. In the latter application this technique is particularly suitable for oral disintegrating tablets since it provides perfect encapsulation at particle level. Spray congealing is very complex technology and it can be affected by many factors, such as working flow rate, coolant type, duress, injection speed and angle, surface characteristics, etc.[2] Improving above parameters, or adding new technologies, will make a great change in thermal dissipation ability. The factors studied were the cooling air flow rate, atomizing force, and molten dispersion feed rate. Dependent variables were the yield, solvability, encapsulation efficiency, particle size, water activity, and flowability.[3] There are some multiple applications of the spray congealing technology: as an example, the encapsulation of milk enzymes, vitamins within the food sector. In the pharmaceutical field, the use of lipoic matrix systems, which can also be produced using this technique, allow determining predictable and stable release performances of active ingredients, is becoming increasingly frequent.[4]

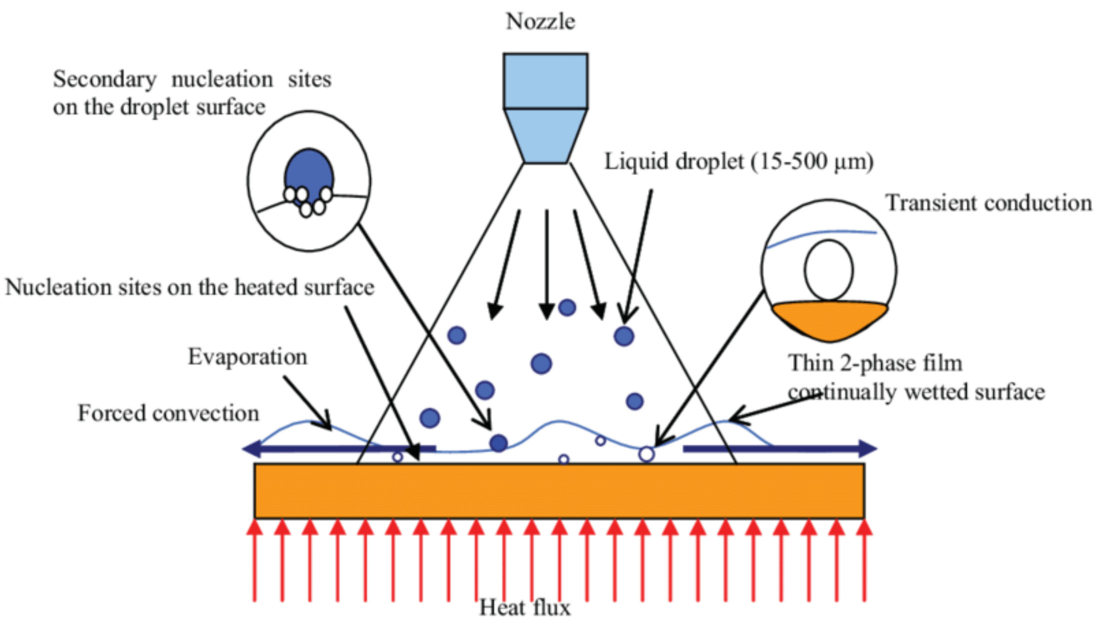


**Fig. 1: The heat transfer mechanisms of spray cooling.[5]**

### Advantages of spray congealing

- Formation of free-flowing microparticles.

- The spray chilling is a technique that consumes less time and energy.
- Spray congealing has high encapsulation efficiency (90–100%) for a lipid matrix composed of solid and liquid lipids.
- Ease of integration into different food matrices, non-toxicity and the probability of encapsulating hygroscopic and moisture-sensitive compounds.
- As per the industrial point of view, it can be performed on an industrial scale.
- It can be adapted for continuous manufacturing.
- Regarding the atomizing equipment and mechanism procedures, spray congealing is closely linked to spray drying.
- Spray drying apparatus can be used for spray cooling processes with some moderation.
- Currently, the use of spray chilling has increased due to the numerous advantages associated with the techno- logical process, such as speed, performance, and relatively low cost.
- Once this method does not need the use of water or organic solvents, the elimination of unused solvents will not be required.
- Also, it is a fast, safe and reproducible physical process because it is associated with an ease adapting of particle size.
- Spray congealing has been considered to be an ecological friendly technique comp ared to other procedures, such as spray drying.
- Spray chilling also lower energy use and time of operation.
- A spare positive aspect is the ease to scale up the production because this technique can be operated continuously with the elimination of some manufacturing steps.
- Additional benefits of spray chilling encompass the utilization of lower temperatures in the procedure, eliminating the need for external heating.
- Furthermore, the controlled release of the SLM contents typically occurs near the carrier's melting point and through the carrier's digestion within the intestinal environment. These SLMs exhibit nearly perfect spherical shapes, contributing to the formation of free-flowing powders.[6,7]

**Process Parameter**

To improve the spray congealing process, different parameters need to be carefully controlled. As may be noted above, the final properties of the dried products are directly influenced by a set of equipment parameters, such as the drying chamber configuration, atomization device and the collector type choice. Additionally, a variety of feedstock specificities and process parameters also play an important role in the final particle feature, conferring different morphologies and residual moisture amounts. It is thus essential to realize how these variables impact the spray-drying mechanism in order to get an optimized operation. There are some of the key process parameters for spray cooling which comes a series of pathways a particle can take towards its yielded shape and size.[8,9,10]

**Atomization pressure-** Nozzle atomizer is used when; atomization stage is carried out under pressure. The chemical properties of the feed material and desired droplet size in turn influence the choice of the atomizer. The force applied during this process effect the droplet size. Higher the pressure, smaller the droplet size of the final product. For a designated atomizer device and feed solution, increasing pressure with decrease in droplet size. According to the mathematical correlation shown below,

$$\frac{Df}{Di} = \left(\frac{Pf}{Pi}\right) - 0.3E8$$

Di and Df are the commencing and concluding droplet sizes when the atomization duress changed from Pi to Pf, individually. In the specific case of rotary atomizers, droplet size shows an inverse relationship with wheel rotation speed and wheel diameter.[6,8,9]

**Feed flow rate-** At a controllable rate, feedstock solution is pumped into the automizer. There is an increase in the droplet size with increasing feed flow rates, when the pressure of atomization kept constant. This is simply understandable bearing in mind that the nozzle would have the same energy amount to expend in the atomization process of higher feeding volumes. Thus, the droplet fissions are minimized, produce a small reduction of its size.[6,7,8]

**Feed viscosity-** The viscosity of the feed is determining that how smoothly it will flow through the spray dryer system. The feed viscosity is enhanced, when a great percentage of atomization energy supplied to the nozzle is used to overcome the large viscous forces of the solution. The motion of the particle relative to one another which develops shear stress, when a liquid moves. Adjoining layers of the fluid tend to move at dissimilar velocities than one another. Hence, a small amount of energy is left for the droplet fission, which results in larger droplet sizes. This mechanism follows.

$$\frac{Df}{Di} = (uf/ui)^{0.2}$$

Di and Df are the initial and final droplet sizes when the solution viscosity exchange from μi to μf, individually. Feed density also follows this principle.[8,9]

**Feed surface tension-** Disruption of the feed surface tension is occur due to atomization. It means that a feedstock solution with higher surface tension block the atomization process. Consequently, before starting the spray-drying process, feedstocks are generally emulsified and homogenized in order to reduce their surface tension.[8]

**Inlet temperature-** The temperature of the drying gas, that measured just before its entry into the drying chamber is known as inlet temperature. The thermal charge of inlet drying gas throw back its capacity to dry the humid atomized droplets and thereby, higher the inlet temperature enables the higher evaporation rate of the solvent. Even now, simply enhance the inlet temperature does not necessarily relent the best final product. Nevertheless, the inlet temperature not only just be increased to achieve better drying performances because, it also has an impact in the wet-bulb temperature of the nearby air. In this process, inlet temperature plays an important role into the shape of the particles at the end. The calculations for level of inlet temperature to be continue depend on the effects of the feed material. Consequently, a wise choice of inlet temperature, balanced on these factors, should be done as maintained by the feedstock properties.[6,8,9,10]

**Drying gas flow rate-** Drying gas flow rate is defined as the volume of drying gas which is injected into the drying chamber per unit time. Movements of particles is increase inside the chamber, due to the high gas flow rate and minimizing air-droplet interaction time. Along with, it is also reported that, efficiency of the cyclone within the drying chamber is increase due to higher the drying gas flow rate. Thus, the gas flow rate should be maintained just sufficient to ensure evaporation of moisture and the separation procedure that follows. This means that the drying gas flow rate should be low sufficient to make sure a complete particle water removal, but on the other hand, it should be suitable for the successive separation procedure.[8,9]

**Outlet temperature-** The temperature of air containing the dried particles, measures prior to deposition of these particles in the collector called as outlet temperature. Apparently, this is the maximum temperature at which the product can be heated. Hence, it does not stand correct in all cases. In case of counter-current dryers, the final product may present a higher temperature than the outlet air.[8,9]

**Residence time inside drying chamber-** Residence time is defined as an exposition period of the atomized droplets inside the drying chamber, being one more main factor with a direct influence on the final product quality. Although, it is basic fundamental to keep the product features and when the dried particles are subjected to longer residence times. Especially upon heat-sensitive materials, thermal degradation may occur. Residence time should be measured accurately to dry the liquid feed keep its core chemical properties. Ideally, the residence time is a few seconds, with fine particles not expend over 15 seconds in the chamber. However, it should be remarked that the residence time is normally in the order of a few seconds. (e.g., in general, fine particles be not to stay more than 10–15 s inside the drying chamber).[8,9]

**Glass transition temperature (Tg)-** Glass transition temperature is a main thermophysical property of a morphos polymers. Above glass transition temperature, the material changes from a rigid glassy state to a rubberier state. For this reason, this could be related anyway with the material stickiness on the drying chamber, being therefore an obstacle to the spray-drying process. Product collection or bunch problems are, for example, one of the major unwanted issues. The glass transition temperature of a feed solution is dependent on its solute constituents. The Gordon-Taylor equation, expresses the Tg of a given feed solution comprise of more than one solute.

$$Tg = \frac{w1 \cdot Tg1 + k \cdot w2 \cdot Tg2}{w1 + k \cdot w2E1}$$

w1 and Tg1 are the weight fraction and the glass transition temperature accordingly of the blend component with the lower Tg. w2 and Tg2 are the weight fraction and the glass transition temperature correspondingly, of the blend component with the higher Tg. k is the ratio of particular heat change of component 1 to component 2 at the glass transition temperature.[8]

**Application-**

Spray congealing is a versatile technique with a wide range of applications in various industries. Its ability to produce solid particles from liquid feedstock through atomization and cooling makes it valuable in many areas. Here are some notable applications of spray congealing.

- **Pharmaceuticals**

Pharmaceutical Industry: antibiotics, medical ingredients, additives, paint pigments, ceramic materials, catalyst supports, and microalgae.[10]

Drug Delivery Systems: Spray congealing is extensively used in the pharmaceutical industry for developing drug delivery systems. It enables the controlled release of active pharmaceutical ingredients, improving drug efficacy and patient compliance. Sustained-release formulations can be created, reducing the frequency of drug administration.[11]

Taste-Masking: Spray congealing can encapsulate bitter or unpleasant-tasting APIs, making them more palatable for patients, especially in the case of pediatric medications.[12]

Solubility Enhancement: This technique can enhance the solubility and bioavailability of poorly water-soluble drugs by incorporating them into amorphous or solid dispersion systems.

**Table No-1: Application of spray congealing to oral bioavailability enhancement of poorly water-soluble drugs used without pre-activation.[13]**

| Drug | Carrier + Additives | Type of SD | Achievement |
|---|---|---|---|
| Carbamazepine | Gelucire 50/13 | Crystalline drug (original polymorph) | Increased in vitro Dissolution rate. |
| Piroxicam | Gelucire 50/13 | Crystalline drug (original polymorph) | Increased in vitro Dissolution rate. |
| Praziquantel | Gelucire 50/13 | Crystalline drug (original polymorph) | Increased in vitro Dissolution rate. |
| Carbamazepine | Gelucire 50/13 | Crystalline drug (change from β to α) | Increased in vitro Dissolution rate. |

1. **Food Industry:** Food Industry: milk powder, coffee, tea, eggs, cereal, spices, flavorings, blood, starch and starch derivatives, vitamins, enzymes, stevia, nutraceutical, colorings, animal feed, etc.[10]

Flavor and Aroma Encapsulation: In the food industry, spray congealing is used to encapsulate volatile flavors, fragrances, and essential oils. This process enhances the stability & shelf life of these compounds, allowing for controlled release in various food products.[6,7]
Nutraceuticals: Nutraceutical ingredients, such as vitamins and probiotics, can be encapsulated for improved stability and controlled release in food & dietary supplements.[14]

- **Chemical Engineering:** In chemical processes, spray congealing can be utilized to produce solid catalysts and specialty chemicals with controlled properties and enhanced reactivity.[13]

- **Cosmetics and Personal Care:** Spray congealing is employed to encapsulate cosmetic ingredients, such as fragrances and active skincare compounds, for prolonged release and enhanced product performance.[14]
- **Agrochemicals:** Pesticides and fertilizers can be encapsulated through spray congealing to enhance their dispersion, stability, and controlled release in agricultural applications.[15]
- **Biotechnology:** Enzymes and biologically active compounds can be encapsulated to protect them from environmental factors and to control their release in various biotechnological applications.[7,16]
- **Textiles:** In textile industry spray congealing used to encapsulate dyes, finishes, and antimicrobial agents to improve their durability and controlled release properties in fabrics. E.g. Wax (high density) and a dye together converted into fine dust.[17]
- **Materials Science:** Spray congealing has applications in producing advanced materials, such as ceramics, nanoparticles, and composite materials with tailored properties.[18]

These applications highlight the adaptability of spray congealing in producing solid particles with tailored properties, ultimately improving the performance and efficacy of products in various industries. The technique's ability to encapsulate and control the release of active ingredients and compounds is particularly valuable, making it a versatile and innovative method for particle production.

## Properties and Characterization of spray congealing

Characterization techniques for spray congealing are essential for assessing the properties of the produced particles and ensuring the quality and performance of the final product. A thorough assessment of the spray congealed SD properties is important as they have a direct impact on the technological and biopharmaceutical behavior of the system. In figure 2 some common characterization techniques for spray congealing are shown which indicates a schematic classification of the applicable MPs properties and of the techniques used for their analysis.[12,19]

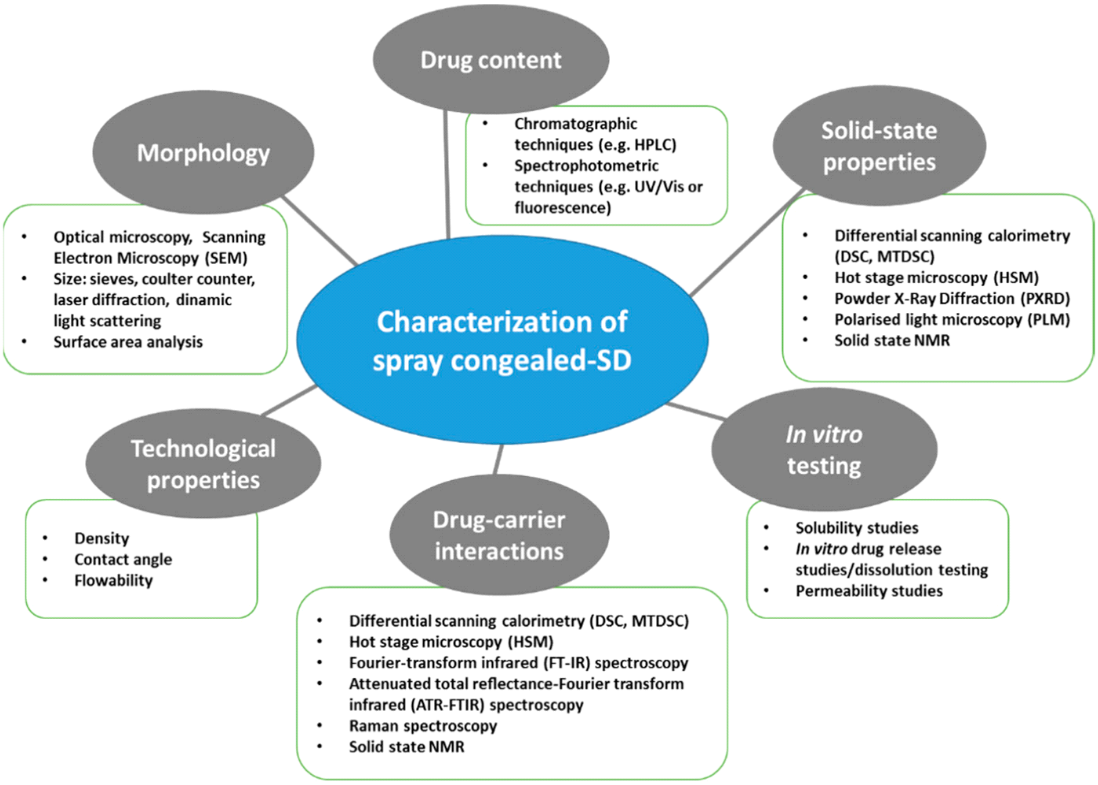


**Fig. 2: Schematic classification of characterization techniques of spray congealing.[19]**

These characterization techniques provide valuable insights into the physical, chemical, and structural properties of spray-congealed particles, allowing researchers and industries to optimize their processes and ensure the quality of the final products.

### Comparative Analysis

A comparative analysis of spray congealing with other particle production techniques helps in understanding its advantages, disadvantages, and areas of specialization. Here's a comparison of spray congealing with two common particle production methods: spray drying and fluidized bed coating.

### Spray Congealing vs. Spray Drying

- Process Overview.

Spray Congealing: In spray congealing, liquid feedstock is atomized and solidified upon contact with a cooling medium.

Spray Drying: Spray drying involves the atomization of a liquid into small droplets, which are then dried by hot air or another gas to produce dry particles.[10,20]

- Particle Characteristics

Spray Congealing: Offers control over particle size, shape, and encapsulation efficiency. Suitable for creating solid dispersions, controlled-release formulations, and taste-masked pharmaceuticals.

Spray Drying: Generates primarily spherical particles with good flowability. Typically used for creating dry powders from liquid solutions or suspensions.[10,21]

- Thermal Stress

Spray Congealing: Less thermal stress on sensitive materials due to lower drying temperatures.

Spray Drying: May subject sensitive compounds to higher temperatures, potentially causing degradation.[14,22]

- Applications

Spray Congealing: Preferred for pharmaceutical applications requiring controlled release and encapsulation of sensitive ingredients.

Spray Drying: Widely used in food processing, ceramics, and the chemical industry for creating free-flowing powders.[10,23]

**Table No-2: Differences between spray cooling and spray drying techniques.[14]**

| Parameters | Spray Drying | Spray Chilling |
|---|---|---|
| Energy flux | Energy applied to the droplets, forcing evaporation of the medium | Energy removed to the droplets, forcing the medium to solidify |
| Equipment | Feed tubes without heating | Heated feed tubes (to prevent solidification) |
| Flow in the equipment chamber | Hot air | Cold air or liquid nitrogen |
| Average particle size | 5–150 μm | 20–200 μm |
| Release mechanism | Dissolution | Difussion, heating |
| Morphology of particle | Particle with irregular geometry and porous surface due to solvent evaporation | Dense, spherical and smooth surface (absence of the evaporation effects of the solvent) |
| Coating | Water-soluble polymers | Waxes, fatty acids, water-soluble and water-insoluble polymers, monomers |
| Food ingredients | Vitamins, flavors, starter cultures, carotenoids, oils and fats, enzymes, acidulants | Ferrous sulphate, vitamins, minerals, acidulants |
| Steps in the process | (1) Disperse or dissolve the asset in the aqueous coating solution<br>(2) Atomization<br>(3) Dehydration | (1) Disperse or dissolve active in the melted lipid mixture<br>(2) Atomization<br>(3) Cooling |

## Spray Congealing vs. Fluidized Bed Coating

- Process Overview

Spray Congealing: Involves atomizing a liquid into a cooling medium to create solid particles.

Fluidized Bed Coating: In this process, solid particles are coated with a liquid or powder in a fluidized bed.[10,24]

- Particle Characteristics

Spray Congealing: Offers control over particle size, shape, and encapsulation. Ideal for creating solid dispersions and controlled-release formulations.

Fluidized Bed Coating: Primarily used for coating existing solid particles with a uniform layer of material.[12,24]

- Encapsulation Efficiency

Spray Congealing: Generally, provides higher encapsulation efficiency for sensitive compounds.

Fluidized Bed Coating: Suitable for creating coated particles but may not be as efficient for encapsulation.[12,25]

- Applications

Spray Congealing: Preferred for applications where encapsulation and controlled release are crucial, such as pharmaceuticals and certain food products.

Fluidized Bed Coating: Commonly used in industries where the coating of granules or pellets is required, such as in the pharmaceutical and agricultural sectors.[14,26]

**Current challenges of spray congealing**

Researchers have considered several factors influencing this interaction to obtain an exhaustive spray cooling correlation and an optimization method to enhance the heat transfer. However, it is challenging to control the experimental parameters owing to the numerous influencing factors.

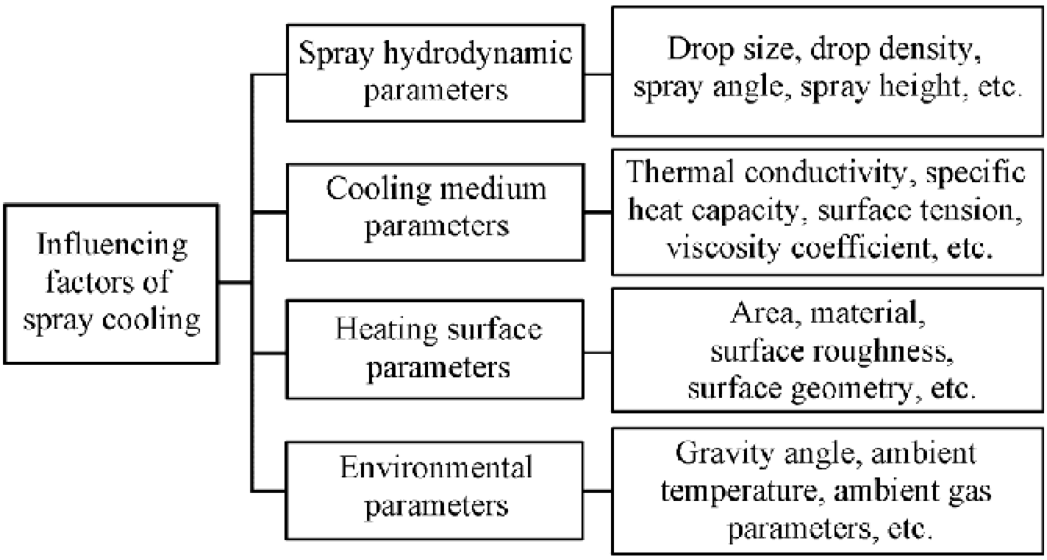


**Fig 3: Influencing factors of spray cooling.[27]**

There are some current provocations of spray congealing technique in the producing of food ingredients when compared to other processes.

- The encapsulating materials of lipid origin that meet the features of an ideal encapsulating agent, such as the oxidative stability, food grade, melting point improvement of the encapsulating structure throughout storage and use, among others.
- Developed a fully hydrogenated milk fat to be used as a surrounding material and describe that fat permit the formation of solid lipid microparticles in the β' form.
- Additional challenges contain the applicability of the microparticles construct by spray congealing in different food matrices, as well as the bioavailability in vitro and in vivo.
- A approaching challenge comprise process extension, as well as optimization in equipment and procedures, to raise the performance in the preparation of solid lipid microparticles.[6]

## CONCLUSION

Spray congealing is an exciting and versatile technique that continues to shape industries, from healthcare to food production. Its ability to finely tailor particle properties make it invaluable for drug delivery and innovative product development. As research and innovation in this field progress, the future of spray congealing is filled with promise, opening doors to new applications and improved products.

This review article aims to provide a comprehensive understanding of the potential and challenges of spray congealing, shedding light on its multifaceted role in modern particle production.

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