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Title	Approaches for improving the flowability of high-protein dairy powders post spray drying - a review
Authors	Hazlett, Ryan;Schmidmeier, Christiane;O'Mahony, James A.
Publication date	2021
Original Citation	Hazlett, R., Schmidmeier, C. and O'Mahony, J. A. (2021) 'Approaches for improving the flowability of high-protein dairy powders post spray drying - a review', Powder Technology, 388, pp. 26-40. doi: 10.1016/j.powtec.2021.03.021
Type of publication	Article (peer-reviewed)
Link to publisher's version	10.1016/j.powtec.2021.03.021
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Item downloaded from	https://hdl.handle.net/10468/13489

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Powder Technology 388 (2021) 26–40

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Review

Approaches for improving the flowability of high-protein dairy powders post spray drying – A review

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ARTICLE INFO

Article history:
Received 30 January 2020
Received in revised form 23 December 2020
Accepted 9 March 2021
Available online 30 April 2021

Keywords:
Powder flowability
High-protein powder
Agglomeration
Granulation
Surface modification

ABSTRACT

Challenges are commonly encountered in the bulk handling and application of high-protein dairy powders, and are strongly influenced by their poor flowability. Powder flowability can be defined as the ability of a powder to flow under set environmental or processing conditions and it is ultimately determined by the type and extent of interparticle interactions occurring in the bulk powder (e.g., van der Waals and electrostatic interactions). High-protein powders are particularly susceptible to the occurrence of interparticle interactions, resulting in increased cohesive forces being experienced in the bulk powder, thereby reducing powder flowability. This review summarises the major factors responsible for poor flowability in high-protein dairy powders and critiques traditional (e.g., agglomeration) and some of the more relevant novel approaches (e.g., dry- and wet-coating and roller compaction) available for improving the flowability of powders post-spray drying. This review material will be of considerable interest to dairy scientists, technologists and engineers challenged with understanding, predicting and controlling the bulk handling and flowability of high-value dairy protein powders.

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Contents

1. Introduction . . . 27
2. Causes of poor flowability of high-protein dairy powders . . . 28
   2.1. Powder bulk composition . . . 29
   2.2. Powder particle and bulk physical properties . . . 29
   2.3. Environmental and processing conditions . . . 29
   2.4. Interparticle interactions . . . 29
      2.4.1. Van der Waals interactions . . . 29
      2.4.2. Electrostatic interactions . . . 30
      2.4.3. Liquid bridging . . . 31
      2.4.4. Solid bridging . . . 31
3. Approaches for improving the flowability of high-protein dairy powders . . . 31
   3.1. Particle size enlargement . . . 31
      3.1.1. Spray dryer and fluid bed agglomeration . . . 31
      3.1.2. Shear agglomeration . . . 33
      3.1.3. Extrusion agglomeration . . . 33
      3.1.4. Roller compaction agglomeration . . . 34
   3.2. Surface modification . . . 35
      3.2.1. Wet coating . . . 35
      3.2.2. Dry coating . . . 35
4. Emerging technologies for improving the flowability of high-protein powders . . . 36
   4.1. Steam jet agglomeration . . . 36
   4.2. Static elimination . . . 37

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https://doi.org/10.1016/j.powtec.2021.03.021
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5. Conclusion and outlook . . . 37
Declaration of Competing Interest . . . 37
Acknowledgements . . . 37
References . . . 37

1. Introduction

Dairy powders are typically produced by dehydrating a liquid milk stream via a combination of evaporation and spray drying technologies in order to increase shelf life and to allow for easier and more economical handling, transport, storage and further application of large volumes of milk solids [1]. As of 2017, the global milk powder market had reached a volume of 9.8 million tons and global market forecasts have predicted this volume will continually increase to approximately 13 million tons by 2023 and to a value of 71.5 billion USD by 2027. The continued commercial growth of dairy powders is attributed to their numerous applications, mostly, but not limited to, the food and beverage industries [2].

Technological advancements have allowed for selective fractionation and enrichment of dairy proteins (caseins and whey proteins), mainly through centrifugal separation and membrane processing (microfiltration, ultrafiltration, nanofiltration and reverse osmosis) of milk streams prior to spray drying. The concentration of dairy proteins, and subsequent depletion of milk serum phase (water, lactose and minerals) and fat constituents can yield a wide range of dairy protein ingredients (Fig. 1). These ingredients can be categorised as either protein concentrates (35–89% protein), e.g., milk protein concentrates (MPC), whey protein concentrates (WPC), micellar casein concentrates (MCC), or as the respective protein isolates (≥90% protein), i.e., milk protein isolates (MPI), whey protein isolates (WPI) and micellar casein isolates (MCI) [3–5]. For the purpose of this review, protein isolates (≥90% protein, low fat and lactose levels) will be the main dairy protein ingredient powder type in focus for further discussion.

High-protein dairy powders have notable nutritional and functional properties, leading to their use in high value commercial applications such as infant and sports nutrition products [6–9]. The value of the global dairy protein ingredient market is expected to reach 58 billion US dollars by 2022. The strong growth of the protein-enriched dairy ingredient market has been driven strongly by the recent consumer demand for high-protein label declarations on a wide range of food and beverage products (e.g., breakfast bars, yoghurts and coffee drinks). In the UK market alone, the appearance of "high-protein" claims has increased by ~500% between 2010 and 2015 [10,11].

The application of high-protein dairy powders requires these powders to be stored in, and discharged from, large silos, transported through pipes (pneumatic conveying) and further processed (e.g., rehydrated, dry blended) in an efficient manner in order to fully harness the beneficial nutritional and functional properties of the powders. The characteristics of these powders i.e., at particle (e.g., particle size, shape and density, surface energy and roughness) and bulk (e.g., cohesive strength, bulk density and interstitial air) levels will determine at what level of ease a powder can be handled and processed for application in varied formulations; this notably includes powder flowability [12].

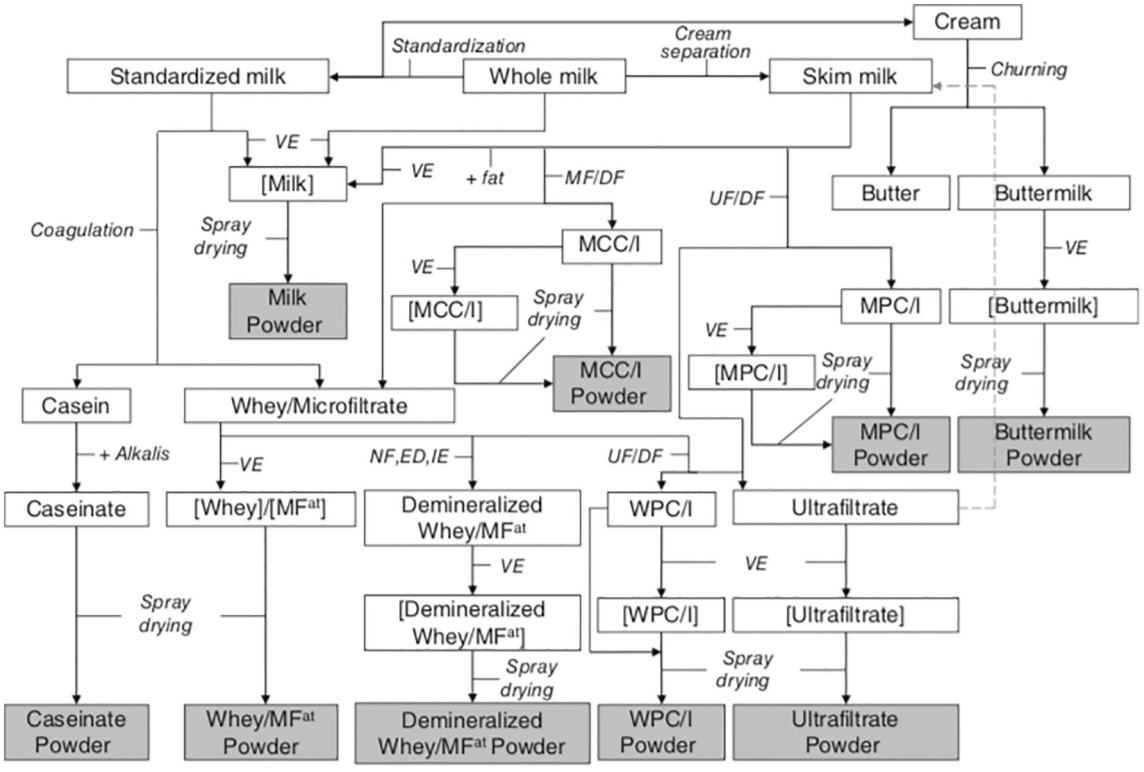


Fig. 1. Fractionation and concentration processes yielding a range of dried dairy powder ingredients (grey boxes) from a liquid whole milk stream.VE: vacuum evaporation, IE: ion exchange, ED: electrodialysis, MF: microfiltration, NF: nanofiltration, UF: ultrafiltration, DF: diafiltration, MFat: microfiltrate. WPC/I: whey protein concentrate/isolate, MPC/I: milk protein concentrate/isolate, MCC/I: micellar casein concentrate/isolate (Schuck, 2013b).

Previously, powder flowability has simply been defined as "the ability of a powder to flow" [13–15]. This definition suggests that flowability is an inherent powder property (i.e., a powder will either flow or not); however, literature has shown that the ability of a powder to flow is ultimately determined by the extent to which interparticle interactions (e.g., van der Waals, electrostatic, liquid/solid bridging) occur, which are heavily influenced by a combination of:

- Powder bulk composition (e.g., fat, protein and moisture contents)
- Powder physical properties (e.g., particle size, shape and distribution, bulk density)
- Environmental and processing conditions (e.g., temperature, pressure and relative humidity).

Studies have shown that even minor changes to the above factors have the ability to cause a marked change in powder flowability [16–18]. Therefore, a more suitable definition of powder flowability is proposed here as "the ability of a powder to flow under set environmental or processing conditions". High-protein dairy powders, such as isolates, generally have poor flowability due to their cohesive nature, making the handling and further application of these powders challenging for manufactures and end-users alike.

During storage in hoppers (e.g., during packaging), cohesive powders, such as dairy protein isolates, have the ability to form structures such as ratholes or arches within the storage hopper, that may act to alter, limit or fully inhibit the flow and discharge of these powders from silos (Fig. 2). Ratholes occur when powder, interacting with surrounding silo walls and other powder particles, becomes stationary and thus creates areas of no-flow within the silo. On the formation of a rathole, powder will continue to flow through a central channel and discharge, although limited, is still possible. The discharge of powder from a silo is completely inhibited when stable arches are formed. Stable arches (also referred to as bridges) may form at the outlet of a hopper when the cohesive strength of a powder is sufficiently high to fully inhibit powder flow. This creates a non-flow regime whereby a discharge aid is needed to re-initiate powder flow from the silo. Routinely, high mechanical force is applied to the outlet of a hopper to encourage discharge of cohesive powders, normally leading to visible damage to the outside of the hopper outlet region [18–20].

For efficient handling after their production, powders must be easily conveyed, normally pneumatically through a series of pipes, sometimes over very long distances within a plant. Cohesive powders, such as dairy protein isolates, have the ability to build up at compact zones of conveying lines such as elbow bends (Fig. 2). At these points, such cohesive powders will collide with a solid surface and cohesive forces can act to cause excessive powder build-up which may cause blockages of powder conveying lines leading to process down time [21–23].

Application of dairy protein isolate powders at industrial scale commonly involves rehydration of these powder ingredients in order to utilise their well-described and often tailored functional (e.g., gelation and surface activity) and nutritional (e.g., delivery of essential amino acids and bioactivity) properties. Efficient rehydration of dairy powders includes the wetting of powder particles, followed by their dispersion and solubilisation. Cohesive powders, such as dairy protein isolates, are prone to form clumps on initial wetting during their rehydration. The formation of these clumps may be attributed to their poor powder flowability as large clusters of cohesive powder particles make initial contact with the surface of the liquid. The subsequent wetting of these clusters occurs only at the surface, forming a gel layer which retards water from penetrating into the clumps of powder particles [24–26].

The challenges outlined above are, in part, caused by poor powder flowability [16,27,28]. The purpose of this review is to comprehensively describe why high-protein dairy powders exhibit poor flowability, as well as to outline the traditional, and to present novel, techniques that are, or may possibly be, utilised by the food industry to improve the flowability of these powders post-spray drying.

2. Causes of poor flowability of high-protein dairy powders

Ultimately, a powder will exhibit poor flowability when the combined cohesive forces between particles (interparticle interactions), and hence the cohesive force of the bulk powder, are greater than the force which is encouraging movement (e.g., gravity for silo discharge and compressed air pressure for pneumatic transport). The types and

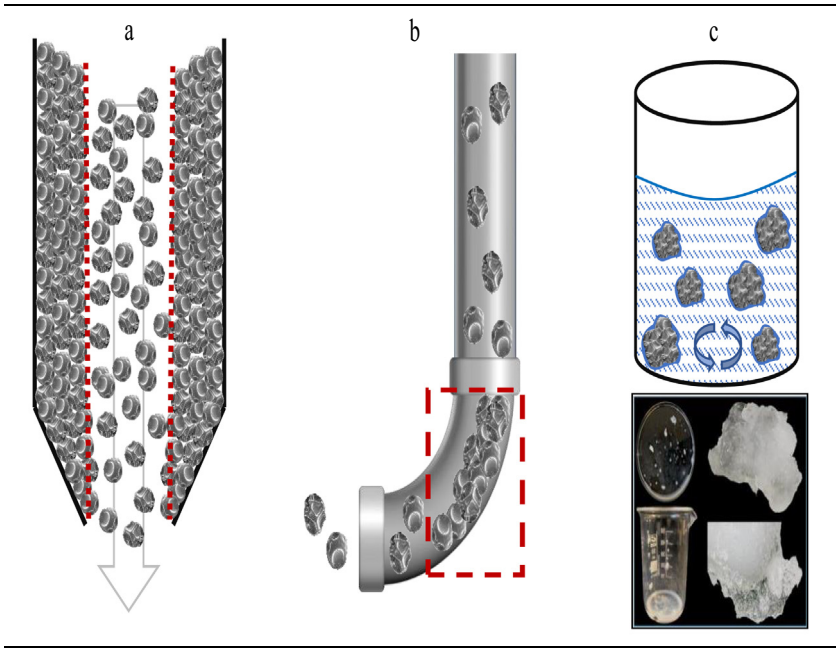


Fig. 2. Schematic representations of challenges arising on the storage (ratholing during silo storage - a), handling (build up on powder conveying line - b) and applications of high-protein dairy powders requiring rehydration (powder clumping on wetting - c).

extent of interparticle interactions occurring are dependent on (i) powder bulk composition, (ii) powder physical properties and (iii) environmental and processing conditions experienced by that powder.

2.1. Powder bulk composition

The extent of interparticle interactions occurring in dairy powders is strongly influenced by these powders bulk composition (i.e., the constituents making up a powder particle), most notably the concentrations of fat [29,30] and protein [31]. Bulk powder composition is ultimately determined by the combination of fractionation and enrichment processes utilised in the manufacture of these ingredients, pre-spray drying (Section 1). For high-protein dairy powders, unit operations such as membrane filtration are utilised to increase the proportion of protein in the bulk composition of the resulting powder (Fig. 1, Table 1). A study by Crowley et al. [16] showed that a significant decrease in powder flowability occurred as the bulk protein content increased for MPC powders with protein content ranging from 35 to 90%. The reduced flowability was attributed to an increased compressibility index on increasing protein content, as the powders with higher protein content had smaller particle size and increased occluded air content, facilitating greater powder particle compaction, yielding increased interparticle interactions (i.e., cohesion) and lower bulk density.

2.2. Powder particle and bulk physical properties

The physical properties of a powder have a large effect on the number and intensity of interparticle interactions occurring between powder particles. These include particle (e.g., size and shape), surface (e.g., area, roughness and energy) and bulk (e.g., density, size distribution) properties [32–34]. These properties are mostly determined by the unit operations and processing parameters utilised during the production of a powder; of example, during powder spray drying, parameters such as atomisation pressure, atomiser type or feed viscosity can be controlled and used to form powders with very different physical properties, leading to greatly different flowability performance [35–37].

High-protein dairy powders usually have smooth surfaced and spherical primary particles, of mean particle diameters less than 100 μm (Fig. 3, Table 1). This results in a bulk powder in which particles can pack together tightly due to their uniform and spherical size and shape distributions, respectively. Also attributable to the small particle sizes is the large surface area per unit mass of powder (Table 1) [16,38–40]. Ultimately, this close packing of relatively uniform, small powder particles yields a bulk powder with high propensity for interactions between powder particles, resulting in strong cohesive forces within the bulk powder, with the resultant powder having poor flowability.

Table 1
Measured values for composition, particle and bulk properties of a representative milk protein isolate powder.

Composition	Protein (%)	85.1 ± 0.8
	Carbohydrate (%)	5.67 ± 0.9
	Fat (%)	1.49 ± 0.0
	Moisture (%)	2.34 ± 0.0
	Ash (%)	5.47 ± 0.1
Physical properties	Particle size (μm)	
	Dv10[a]	11.9 ± 0.3
	Dv50[b]	31.7 ± 0.7
	Dv90[c]	64.7 ± 0.2
	Sphericity[d]	0.74 ± 0.0
	Specific surface area (m^2kg^{-1})	212 ± 4.4
	Particle density (g cm^{-3})	1.38 ± 0.0
Bulk properties	Bulk density (g cm^{-3})	0.27 ± 0.0
	Tapped density (g cm^{-3})	0.43 ± 0.0

[a] Dv10 - Particle size below which 10% of material volume exists.
[b] Dv50 - Particle size below which 50% of material volume exists– median.
[c] Dv90 - Particle size below which 90% of material volume exists.
[d] Sphericity - Unitless value representing sphericity of a particle (1- perfect sphere).

2.3. Environmental and processing conditions

The relative humidity of the environment surrounding a high-protein dairy powder will impact the nature and extent of interparticle interactions occurring in the bulk powder. Studies have shown that at high relative humidity, the moisture surrounding powder particles condenses at the contact points and thus, through capillary action, creates a liquid bridge (Table 2). The reduction of flowability through liquid bridge formation is prominent in powders containing large amounts of hygroscopic materials (e.g., carbohydrates). Dairy protein isolates tend to contain low proportions of lactose (and other carbohydrates) and thus, a very high relative humidity (~ 80% RH) is needed in order to significantly reduce their flowability through caking [17,41–45].

Relative humidity also impacts the surface energy of powder particles, impacting bulk powder flowability. Dairy protein isolates, like other high-protein dairy powders are strongly hydrophobic due to the presence of protein and fat at the surface of the powder particles [46–48]. Previously, Karde et al. [49] showed that while on increasing relative humidity, the surface energy of hydrophilic powders also increased, no change in surface energy was experienced on the same increase in relative humidity for ibuprofen which, like protein isolates, is highly hydrophobic.

The extent of consolidating stress acting on powders is another well studied factor influencing flowability of powders [16,50]. Hoppers are often used for short-term storage of MPI and in such intermediate hoppers, varying pressures (termed head pressure) act downward on the powder particles, to an extent dependent on hopper dimensions/capacity, extent of fill and exact position of powder particles within the hopper, all together resulting in a bulk powder exhibiting differing flowability behaviour. On exceeding a threshold pressure, a bulk powder will experience either deformation, which encourages flowability (e.g., low bulk density powders, such as MPI) or consolidation, which decreases flowability (e.g., high bulk density powders such as skim milk powder) [51]. This consolidation-induced reduction in flowability is caused by plastic deformation at contact zones between powder particles, resulting in increased number and strength of interparticle interactions, thereby increasing the cohesive bulk strength [52].

2.4. Interparticle interactions

The main attractive forces occurring between powder particles are van der Waals, electrostatic, liquid- (via capillary forces) and solid-bridging interactions, and are summarised in Table 2 [53]. For dairy powders with large mean particle size (>150 μm), such as agglomerated powders, the forces acting upon these powders, such as gravity, usually outweigh these attractive forces and thus, such powders tend to flow well. For powders with smaller mean particle size (<100 μm), such as dairy protein isolate powders, the number and extent of the above attractive interactions may outweigh the effect of the acting forces, hence causing powder particles to adhere to each other, reducing powder flow [14,53].

2.4.1. Van der Waals interactions

Van der Waals interactions are known to have one of the lowest adhesion strengths of those presented in Section 2.4. However, they are the predominant force causing adhesion in dry powder systems of small particle size, such as protein isolates, due to the large number of van der Waals interactions occurring at the particle level in these powders [14,54,55]. Van der Waals interactions occur as electrons are in constant movement in powder particles, leading to the continual formation of temporary dipoles at the surface of (and throughout) powder particles. A dipole is a set of charges, of equal magnitude, but opposite

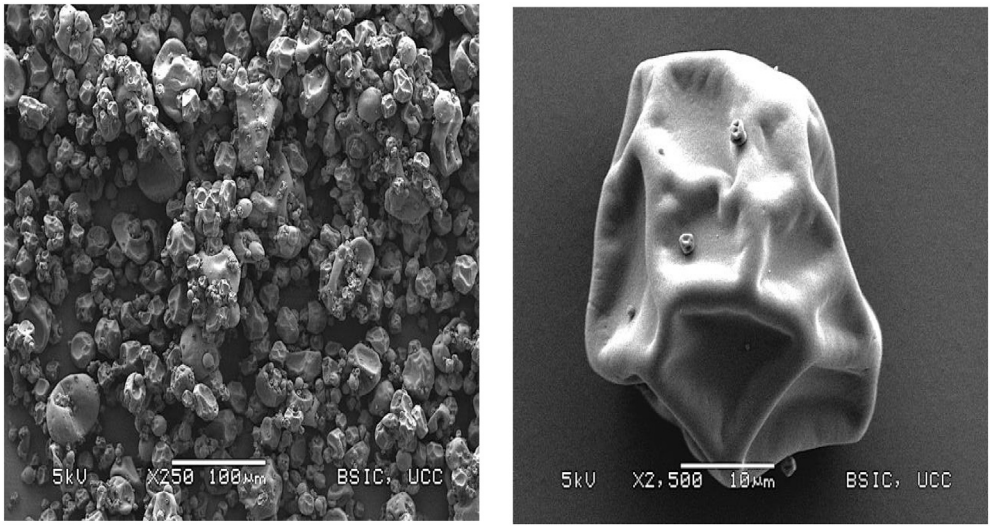


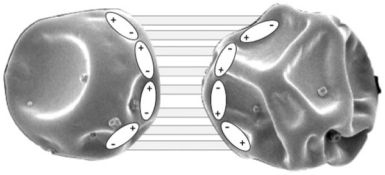
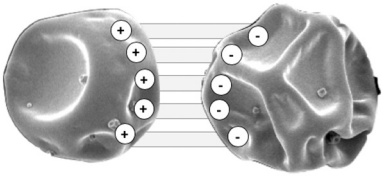
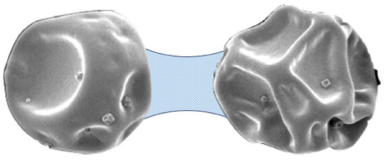
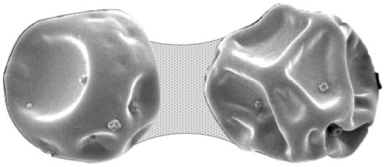
Fig. 3. Scanning electron micrographs (magnifications- × 250 and × 2500, scale bars of 100 μm and 10 μm included) of a representative milk protein isolate powder.

sign (Table 2), and thus, the adhesion of two (or more) powder particles occurs due to the attractive forces that exist between the dipoles of opposite powder particles (via ionic bonding) [53,56]. It is well established that the most influential powder particle and bulk properties for determining the amount of van der Waals interactions occurring are particle size and interparticle spacing, respectively [57–59]. Commercial dairy protein isolate powders are composed of primary particles of small particle size (<100 μm) which can pack together closely due to their spherical shape, and these features align closely to the conditions needed for van der Waals interactions to occur to a great extent in the powder bulk, thereby limiting flowability of high-protein dairy powders.

2.4.2. Electrostatic interactions

The forces associated with electrostatic charging (also termed "tribocharging") are, again, of low strength when compared to other interparticle forces [54]. Like van der Waals interactions, electrostatic charging can also have a large effect on the flowability of high-protein dairy powders due to the extent to which they can occur (especially when combined with van der Waals interactions). During the production of dairy powders, particles frequently collide with solid surfaces (termed particle collisions) or with other powder particles (termed particle attrition), during processes such as pneumatic conveying, fluid bed processing and dry blending. On such collisions, the surface of dairy

Table 2
Summary of interparticle interactions occurring at particle level in high-protein dairy powders. Scanning electron microscopy micrographs of milk protein isolate powder particles used to represent powder particles.

	Description	Illustration	Strength	Magnitude
Van der Waals	Temporary **dipoles** at powder particles interact via ionic bonding with dipole of **closely located particles**.		+	++++
Electrostatic	Opposite **charges** on surfaces of powders from **friction and attrition** lead to ionic bonding between particles		++	+++
Liquid bridging	Surrounding or internal **moisture condenses** at contact points between particles. **Liquid capillary forces** cause adhesion.		+++	++
Solid bridging	**Solidification of liquid bridges** via evaporation or crystallisation cause large increase in adhesion strength.		++++	+

powder particles builds a charge, and due to the organic nature of dairy powders, insulation of this charge will occur, leading to a very slow dissipation of charge over time. Oppositely charged particles, if in a close arrangement (such as the case for dairy protein isolate powders), will create attractive forces that lead to the cohesion of the particles, limiting powder flowability [53,60,61].

2.4.3. Liquid bridging

It is possible for physical bridges to form between two or more powder particles, and the resulting interparticle interactive forces can be strong enough to considerably reduce the flowability of dairy powders [53,54]. In addition to van der Waals and electrostatic interactions, the physicochemical properties of a dairy powders, such as particle size and shape, also play a role in the formation of these interparticle bridges. Environmental conditions surrounding the powder, such as relative humidity and temperature, also strongly influence the formation and stabilisation of these interparticle bridges [42].

Liquid from within powder particles (moisture or liquid fat) or from the environment (i.e., relative humidity) may condense at contact points of closely located particles, with this condensate creating many cohesive liquid bridges (termed pendular bridges) between particles due to the presence of a strong attractive capillary force which is reported to be over 50-times stronger than van der Waals forces [54,62,63]. The occurrence of liquid bridging during the storage of dairy protein isolate powders is low compared to other commercial dairy powders (e.g., whole milk or skim milk powder), which may be attributed to the much lower level of hygroscopic carbohydrates present in dairy isolate powders due to the protein fractionation/enrichment (i.e., lactose reduction) steps in their production (Fig. 1, Table 1).

2.4.4. Solid bridging

Under appropriate conditions (e.g., temperature and relative humidity), a liquid bridge may be further stabilised through transition into a solid bridge via evaporation, crystallisation, or solidification of the bridging material, thereby increasing the strength of attractive forces between particles [66,67]. If occurring in an uncontrolled manner (i.e., caking), the formation of liquid or solid bridges will significantly reduce the ability of dairy powders to flow and make the handling and storage of these powders more challenging. In contrast, the controlled formation of a liquid and subsequent, solid bridge, is routinely utilised as a means of particle size enlargement in order to control powder flowability which will be the focus of later sections of this review (e.g., Section 3.1).

3. Approaches for improving the flowability of high-protein dairy powders

Due to the growing market for high-protein dairy powders, there is a continual need to tailor and control the flow properties of such powders, in order to ensure easy storage, handling and further application. To increase a powder's flowability, actions must be taken in order to reduce the number and extent of the above interparticle interactions from occurring at the bulk level. Traditional and more novel approaches to control a powder's flowability exist and are utilised by various industries handling powders such as the food, pharmaceutical and biochemical industries, and these approaches may be categorised as (i) particle size enlargement and (ii) surface modification of powder particles.

3.1. Particle size enlargement

The most traditional approach to increase a powder's flowability is to increase the size of the particles that make up a powders bulk, allowing the forces encouraging movement (such as gravity or pressurised air) to outweigh the forces of cohesion, thereby, allowing the powder particles to flow. Inversely, the extent of interparticle interactions will be reduced, serving to further improve powder flowability [19]. The terms agglomeration (mainly used within the food industry) and granulation (mainly used within the pharmaceutical industry) are both used to describe the processes of increasing the particle size of a powder by combining numerous individual primary powder particles together into large cluster-like structures where the individual particle may still be distinguishable. These clusters of particles are of a much greater size than that of the original particle, thus increasing flowability. Agglomerated particle structures contain increased volumes of interstitial air in the form of capillaries and pores, reducing the powder's bulk density and allowing for easier penetration and movement of water into and through the cluster structures through capillary action on rehydration [35,68,69]. For the purpose of this review, the term agglomeration will be used to collectively describe the various methods for particle size enlargement that follow.

3.1.1. Spray dryer and fluid bed agglomeration

The process of agglomeration in the production of dairy powders generally occurs in the spray dryer chamber, and/or, in the external fluid bed located after the spray drying chamber (Fig. 4) [70,71]. In the spray drying chamber, agglomeration occurs through the collisions of either wet particles (primary agglomeration) or wet and dry particles (secondary agglomeration). To some extent, both primary and secondary agglomeration occur naturally in the spray chamber through random collisions of moving particles in the chamber (spontaneous agglomeration). However, by controlling the geometries and parameters of the spray drying process, both primary and secondary forms of agglomeration can be forced to occur to varying extents (forced agglomeration) [35,72,73]. Fine powder material, produced during the drying process may be recirculated back into the top of the spray dryer main chamber and directed at the atomisation zone, where new primary particles are being formed, allowing the dry fine material to collide and coalesce with newly formed, wet particles leading to the formation of large porous clusters of particles with channel spaces between the individual primary particles (Fig. 4). The degree of agglomeration via fines return may be controlled by altering the amount of the fine powder particle material that is recirculated back into the dryer main chamber [1,15,74].

On leaving the spray drying chamber, powder particles generally enter into an external fluidised bed unit where heated and cool air pass upwards though the powder separately, at velocity, to complete drying and cooling of the powder, respectively. This high velocity air pushes powder particles upwards and disperses them into the chamber of the fluid bed, i.e., fluidising them in order to finalise their drying and facilitate subsequent cooling (Fig. 4) [58]. Particle size enlargement of the powder through fluid bed agglomeration can also be achieved here by rewetting, i.e., applying a binding solution onto the particles as they enter into the fluid bed unit. The process in which the binder acts to agglomerate the powder particles has been well studied and is separated into four main stages: binder addition, wetting and spreading of the binder, nucleation and solidification (Fig. 4) [68].

3.1.1.1. Binder addition. The method of addition of the binding solution may vary from pouring to pumping, but studies have shown that the most uniform agglomeration process (large agglomerates of a narrow particle size distribution) is achieved by atomising the solution onto the powder particles as they enter the fluid bed unit [75]. A twin-fluid nozzle is routinely used to atomise the viscous binder solution using high air pressures to form a uniform spray of the solution in droplet form which can come into contact with the powder particles.

3.1.1.2. Wetting and spreading. When the binder first comes into contact with the powder particles, the binder will begin to wet the surface of the particles. Due to the fluidisation movement of the particles, the binder will spread and coat the entire surface of the powder particles, which is important for the formation of agglomerates of a uniform particle size.

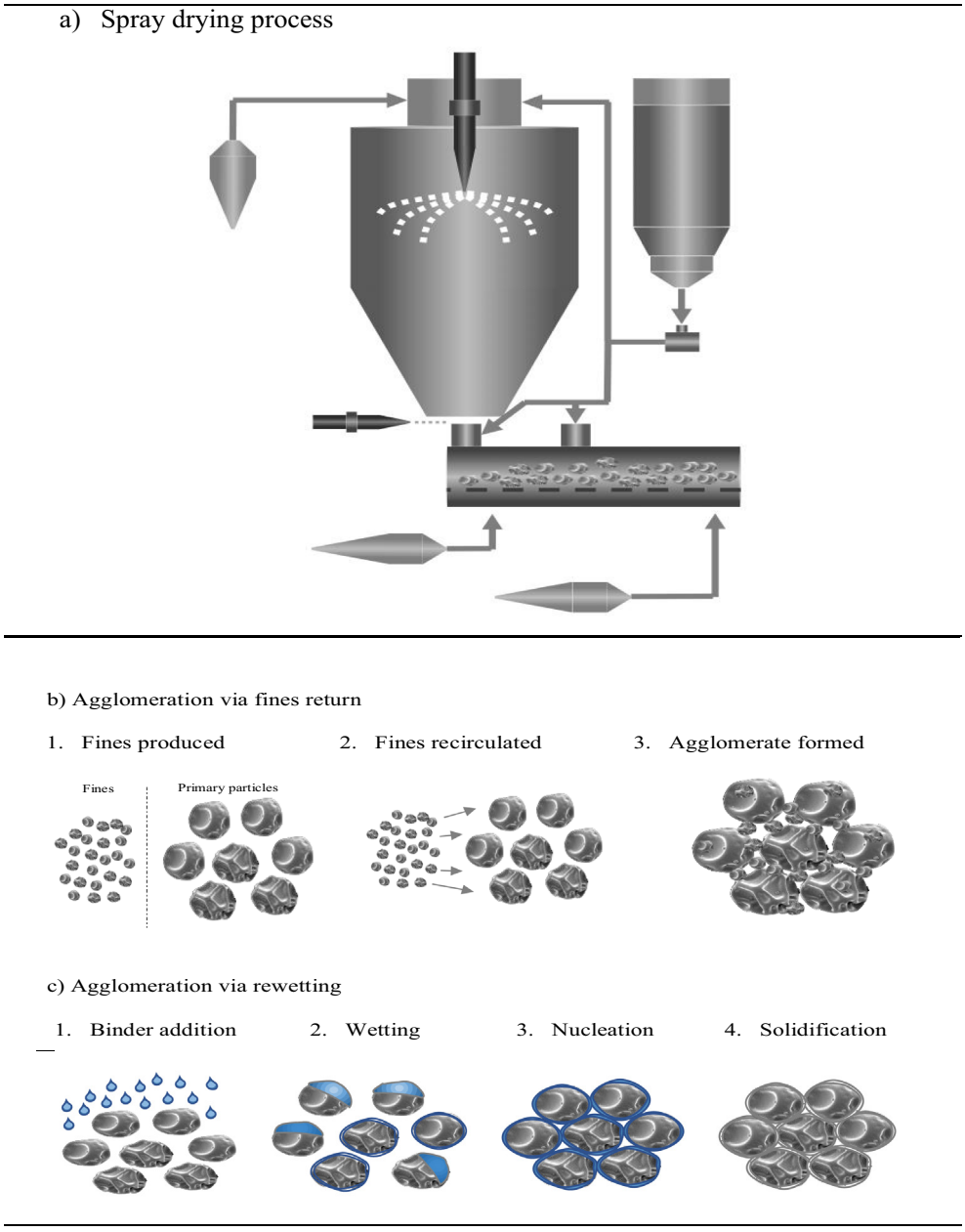


Fig. 4. Schematic representations of (a) the spray drying process used for dairy powders with external fluid bed drying and (b) agglomeration via fines return and (c) agglomeration via rewet processing.

3.1.1.3. Nucleation. Initially, collisions of the now-wetted particles cause the formation of liquid bridges (Section 2.4.3), which link two or more particles together through capillary forces. This initial particle growth acts as a nucleus for further consolidation and growth of the agglomerate structure.

3.1.1.4. Solidification. Due to the elevated temperatures (approx. 70 °C) of the initial stages of the external fluid bed, the liquid bridges stabilising the agglomerate structures will be, partially or fully, evaporated, crystallised or solidified depending on the compositional make-up of the liquid bridge. Solidification of the liquid bridge leads to the formation of a strong, solid bridge between primary particles in an agglomerated structure.

The binding solution used for fluid bed re-wet agglomeration of powders can vary depending on the application, with binder characteristics such as composition, viscosity, rate of addition and surface tension all playing vital roles in determining the type, size and strength of agglomerates formed. Water and sugar solutions are some of the binders used by various powder handling industries during fluid bed agglomeration; however, for the dairy industry, lecithin, a natural, phospholipid-rich surfactant which has been traditionally used for coating agglomerates formed via fines return, has been identified as a potential binder for powder agglomeration due to its well established beneficial impact on the rehydration properties of the resulting agglomerated structures (sometimes termed lecithination/instantisation) [76,77]. Lecithin, usually dispersed in a blend of vegetable oils, is highly viscous and can effectively bind numerous particles together in large cluster-type structures. Due to its high fat content, at the elevated temperatures of the external fluid bed, the lecithin blend can efficiently spread over the surface of the powder agglomerate structure, which is then solidified on cooling. As lecithin is rich in phospholipids, by coating the surface of an agglomerated powder particle it in turn increases the

hydrophilic nature of the powder surface due to the presence of the amphipathic, phospholipid molecules which has a very beneficial impact on the wettability of the resulting agglomerates [9,78].

The agglomeration of high-protein dairy powders via spray dryer and fluid bed agglomeration is not widely practiced by dairy industries. Although particle size enlargement has been clearly shown to improve the flowability of many dairy powders it must be noted that it does this by altering the physical and bulk properties of the original powder. For example, agglomerating a high-protein dairy powder with the aid of lecithin (or other binding solutions), will reduce concentrations of other constituents of that powder [80]. This must be avoided in the production of high-protein dairy powders such as protein isolates (MPI, WPI) where strict requirements for protein content of the final powder (≥ 90% protein) must be satisfied. Also, when high-protein dairy powder is agglomerated, the bulk density of that powder decreases, meaning that the same mass of the agglomerated powder will occupy a larger volume than that of the non-agglomerated form. [81] showed that for MPI, the initial low bulk density (0.30 g cm^{-3}) of the powder was further reduced (0.18 g cm^{-3}) on agglomeration by fluid bed processing. These effects of spray dryer and fluid bed agglomeration can be seen as a disadvantage for the dairy powder industry as it can lead to a reduction in product value and an increase in storage/transport costs, respectively.

Studies by Gaiani et al. [82] and Mimouni et al. [83] have shown that casein- and whey-dominant powders (e.g., MPI and WPI, respectively) have different rate limiting steps in their rehydration processes and therefore, agglomeration of these powders may yield different effects on their rehydration behaviour. For whey-dominant powders, the initial wetting stage is the rate limiting step whereas, for casein-dominant powders, particle dispersibility limits the rehydration. As particle size enlargement via spray dryer and fluid bed agglomeration effects the wetting stage of rehydration, it can positively impact the rehydration of whey-dominant protein powders, such as WPI, whereas it can negatively impact the rehydration of MPI and MCI powders, which are high-protein, casein-dominant powders; the clusters formed on agglomeration of MPI/MCC powders break down more slowly in solution, negatively impacting powder dispersibility and solubility [84]. Ultimately, a more comprehensive approach to improve the flowability of high-protein dairy powders is required which minimises any negative effects on the compositional and rehydration properties of these powders.

3.1.2. Shear agglomeration

Shear agglomeration is a method of particle size enlargement, commonly used in the pharmaceutical industry. The process shares many similarities to that of fluid bed agglomeration (Section 3.1.1) as again, a binding solution is used to facilitate the agglomeration of primary particles into larger structures. The binder is added to a mass of powder that is in constant movement and shear mixing in an agglomerating drum/vessel. On continual mixing in the presence of the binder solution, the powder particles form relatively large agglomerates (>1 mm in size) through surface wetting, nucleation and consolidation. Agitators in the agglomeration device mix at specific speeds to introduce shear forces which act to reduce the particle size of the formed agglomerates towards a smaller target agglomerate size through the forces of attrition and breakage [85–88].

The level of shear used can vary (low or high shear) but most routinely high shear forces are used to treat the agglomerating powder particles. Litster and Ennis [89] report that for high shear agglomeration, the mixing vessel will be rotating at 60–800 rpm, while the agitator provides high levels of shear by rotating at 500–3500 rpm typically. The shear forces experienced during agglomerate formation causes densification, whereby the particles that make up an agglomerate are further compressed, minimising interparticle channels and causing the entrapped binder to be squeezed out to the surface of the agglomerated structure, allowing for further nucleation and agglomeration to occur. The resulting agglomerates of shear agglomeration therefore, are quite often large, dense structures that are spherical in shape [90]. The effect of agglomerate densification on the resulting powder bulk density during shear agglomeration processing was illustrated in a study by Chevalier et al. [91]. Here agglomerates of calcium phosphate were formed via low and high shear agglomeration. It was found that the agglomerates formed using high shear forces had a much higher bulk density (0.73 $g.cm^{-3}$) than that of the agglomerates formed using low shear forces (0.53 $g.cm^{-3}$).

For the agglomeration of high-protein dairy powders, shear agglomeration provides no clear advantages to that of the current dairy industry standard, fluid bed agglomeration. The formation of more dense agglomerates may alleviate the reduction in bulk density experienced on fluid bed agglomeration; however, this densification process acts to eliminate the interparticle channels and pore spaces between particles, with these components of agglomerated dairy powders being key in the efficient wetting on rehydration; interparticle channels allow the surrounding liquid to easily penetrate into inner primary particles of the agglomerated powder. Large, dense agglomerates of high-protein dairy powder particles could lead to further clumping issues on powder wetting (Fig. 2).

3.1.3. Extrusion agglomeration

Extrusion technology is regularly used for the continuous agglomeration processing of pharmaceutical powders at commercial scale. Another form of wet agglomeration, extrusion agglomeration utilises water or a more complex binder solution to cause the agglomeration affect. For the agglomeration of hydrophobic or poor wetting powders, such as high-protein dairy powders, the adequate and efficient distribution of the binder, to allow for uniform nucleation and growth, is a limiting process step. Extrusion agglomeration exposes powders to high forces through intense mixing and kneading in the extrusion line which forces the spreading of the binders across the powder particles allowing the size of agglomerates to increase [69,92].

In the extrusion agglomeration process, the powder drops, or is fed, into a barrel where screws counter-rotate at a set speed; the number of screws varies, with the most common configuration being a twin-screw extrusion line. The counter-rotating motion of the screws transports the powder along the extrusion barrel and creates a mixing motion in the bulk powder. A liquid feed line is used to introduce the selected binder, at a pre-determined addition rate, into the extrusion line, and into contact with the powder forming liquid bridges. As the wetted powder particles pass through the barrel, they enter kneading zones where discs of specific geometries act to create zones of high shear forces. These forces, as in high-shear agglomeration, cause densification of the agglomerated particles and allow for further agglomerate growth. Each section of the extrusion agglomeration process allows for heating, drying and cooling of the agglomerated structures as the extrusion barrel is jacketed allowing for temperature control. Many process geometries (e.g., number of screws, screw length and diameter, kneading elements) and parameters (e.g., screw speed, liquid:solid ratio) may be utilised in order to control the extrusion agglomeration process and thus, tailor the physical properties of the resulting agglomerated powder, as discussed at length by Seem et al. [92]. The final step in the process is the shaping or milling of the formed agglomerate (extrudate) to the desired particle size/shape. In the pharmaceutical industry, the extrudate is commonly compressed directly into tablet form which is a widely used form of powder delivery. However, if implemented in the production of high-protein dairy powders, a milling step may be utilised to control the particle size of the agglomerates formed in the extrusion process (Fig. 5) [93–95].

In a series of studies, Keleb et al. [96,97] used pharmaceutical powders that exhibit poor flow and dissolution properties (i.e., α-lactose monohydrate and paracetamol), to evaluate the efficiency of an extrusion agglomeration process with (polyvinylpyrrolidone as binder), and without (water as binder), the use of complex binding solutions,

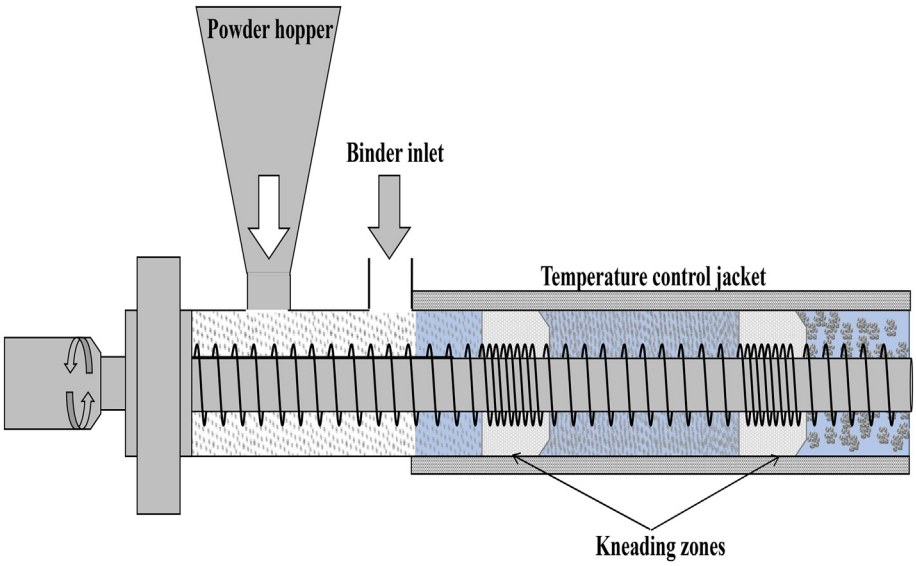


Fig. 5. Schematic representation of a typical extrusion granulation process.

in direct comparison to a shear agglomeration process. The results showed that agglomeration was achieved for all powders without the use of complex binding solution (polyvinylpyrrolidone) for extrusion agglomeration (high agglomerate yield and over 60% of the agglomerates formed had particle size greater than 250 μm) while the agglomeration of these powders was not possible using shear agglomeration without the use of polyvinylpyrrolidone as a binder. The possibility of agglomerating a difficult to handle powder, such as paracetamol, with only water suggests that this approach to particle size enlargement may be attractive in the processing of high-protein dairy powders.

3.1.4. Roller compaction agglomeration

All approaches for particle size enlargement discussed thus far in this review have been forms of wet agglomeration, where binding solutions are used to facilitate the agglomeration of powder particles. On the other hand, roller compaction is a form of dry agglomeration where particle size enlargement is achieved by applying very high levels of pressure which forces particles closer together and induces changes in the particle structures, yielding agglomerates. Dry granulation is routinely used in the agglomeration of powders that are sensitive to moisture and therefore cannot be processed via traditional wet agglomeration approaches. Roller compaction is the most studied, and applied, form of dry agglomeration, commonly used in the pharmaceutical industry to improve the flowability of various pharmaceutical powders. The clear advantages to dry agglomeration (e.g., no binder needed, no heating or drying steps required) highlights it as a method of particle size enlargement with potential in the production and control of difficult to handle high-protein dairy powders [93,98].

A typical roller compaction process involves feeding a powder, either via gravity (for powder with good flowability) or a screw feeder (for powder with poor flowability, such as high-protein dairy powders) into the contact zone of two large co-rotating rollers. At this point, termed the "nip region", large frictional shear forces will draw the powder down and between the two rotating rollers. Very large pressures begin to build up within the powder bulk which causes two main conformational changes in the powder particles. Firstly, particles are forced to rearrange into close arrangement, and then, as the pressure continues to build, a critical point for the particles is met, which either causes particle breakage (for fragile particles) or particle deformation (for stronger particles). As a result, the powder exits the rollers in the form of heavily compacted ribbon-like structures that can be gently milled and screened to form agglomerated powder structures with desired particle sizes (Fig. 6) [99–101]. The ability to produce dense granulated structures of MPI with controlled particle size post-milling, poses a unique opportunity to modify the bulk handling properties and applications of high-protein dairy ingredients where rehydration is not required in a final application. In addition, the initial compression into ribbon forms could support applications of MPI into new markets such as milk candy which are growing in popularity in certain geographies (e.g., Asia).

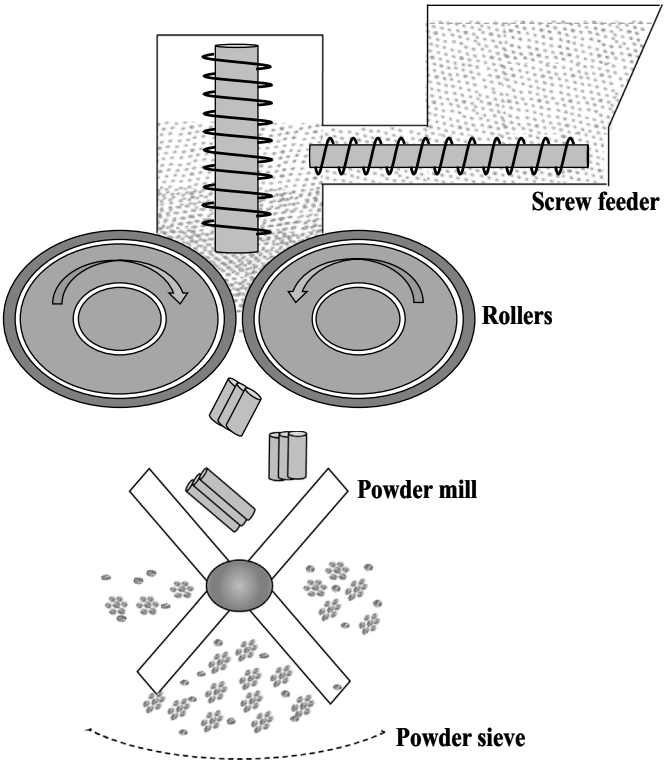


Fig. 6. Schematic representation of the roller compaction agglomeration process for dry powder followed by milling to achieve the desired powder particle size.

3.2. Surface modification

The surface characteristics (e.g., surface energy and charge) and composition (e.g., surface fat) of dairy powders plays a key role in determining the number and extent of interparticle interactions occurring in the powder bulk, and thus, impacts the powder's flowability (Section 2.4). For high-protein dairy powders, due to their small and spherical size and shape, the surface properties of these powders allow for extensive van der Waals and electrostatic interactions to occur, leading to such powders having high cohesive strength. The presence of fat at the surface of powder particles is also attributed to reduced powder flowability. During production of dairy powders by spray drying, fat contained in the atomised droplet will be preferentially located at the powder particle surface on atomisation and subsequent drying, leading to an over representation of fat at the surface when compared to bulk of the powder [102–105]. Even in high-protein dairy powders, where fat usually contributes to a very small percentage of the total bulk composition (approx. 1–5% fat), the surface composition of these powders may be dominated by fat. Studies by Kim et al. [12,106] focussing on the effect of surface composition on the flowability of spray dried dairy powders showed that for a WPC 80 powder (86% protein, 6% fat), over 50% of the exposed powder surface was comprised of fat, despite the fact that fat constituted only 6% of the bulk powder composition. This over-representation of fat at the surface of dairy powders, compared to the bulk, also occurs in casein dominant systems such as MPC, MPI and MCC [107,108]. The presence of fat at the surface of powders is a strong factor influencing (generally adversely) their flowability and rehydration properties. In the case of high-protein powders, where small, spherical particles are in close arrangement, the presence of fat at powder surfaces may play a critical role in large cohesive strength experienced in the bulk powder, and ultimately act to limit their flowability.

Surface modification is a method of processing which acts to alter the surfaces of powder particles by creating a new powder surface or shell in order to improve the bulk handling characteristics of powders. By altering only the surface of a powder particle, changes in particle size and shape are minimised and thus, as are large changes to the bulk density of a powder. Minimising the decrease in bulk density (a feature of particle size enlargement - Section 3.1), is desirable in the production of high-protein dairy powders for storage and transport costs. Two main methods of surface modification have been previously utilised by both food and pharmaceutical powder processing industries, and these are fluid bed coating and dry coating of powder particles.

3.2.1. Wet coating

Wet coating (also termed fluid bed coating) of powder particles via fluid bed processing utilises powder fluidisation and liquid atomisation to achieve a very thin layer of coating material on the surface of a powder. This newly formed layer is then dried rapidly and cooled in a manner that avoids particle agglomeration, creating a new powder boundary that alters the surface properties and composition to improve powder flowability. Surface coating has been utilised in food applications previously using biopolymers such as lactose, glucose, starch hydrolysates and alginate solutions as coating solution for food powders. Close control over the composition and viscosity of these coating solutions, as well as the addition level and rate is necessary to achieve uniformly coated particles while preventing unwanted agglomeration during fluid bed coating of powders. [109–111].

Fluid bed coating utilises the same principles as fluid bed agglomeration (Section 3.1.1), where a liquid is atomised onto particles in fluidised motion. However, a different geometry of fluid bed, termed a "Wurster coater", is used for particle coating applications to avoid the unwanted agglomeration of particles in this process. Here, a central channel is inserted at the bottom of the fluid bed chamber which acts to accelerate and disperse the powder particles upwards and through an atomisation zone where a thin layer of the coating solution is applied to the particle surface. The nozzle used for atomisation of the coating solution is loaded from the bottom of the fluid bed chamber (bottom-fed atomisation) and it sits in the central channel of the Wurster coater, spraying very fine, uniform droplets of the coating solution into the atomisation zone of the Wurster coater. As the particles become coated, the high velocity air continues to push them upwards into the fluid bed chamber in a fine dispersion. As the coated particles exit the Wurster coater's inner channel, they enter into the top of the fluid bed chamber where the elevated internal temperature rapidly acts to dry and solidify the newly formed particle coatings, before the particles are cooled and collected (Fig. 7) [112–114].

For efficient particle coating while minimising unwanted agglomeration, each step of the above process must be carefully controlled, most notably, the fluidisation of particles into a fine dispersion where particle-particle interactions and collisions are avoided. Geldart [115] developed a classification system to characterise the ease, or difficulty with which a powder can be fluidised, describing "Group C" powders (C – cohesive) as the most difficult to fluidise due to their characteristic low particle size and low particle density, both being important properties of high-protein dairy powders (Fig. 8). This indicates that the efficient coating of high-protein powders may prove difficult due to improper fluidisation of high-protein dairy powders (e.g., channelling/ratholing in powder bed) [116]. Chen et al. [117] reported that unwanted agglomeration is unavoidable when coating powder particles with sizes ≤50 μm (typical of high-protein dairy powders) without prior modifications such as pre-treatment of powder via dry coating (Section 3.2.2) or though modified fluidisation (e.g., vibro-fluidisation).

3.2.2. Dry coating

Traditionally, wet coating of powder particles was the standard procedure for surface alteration to tailor the flowability and fluidisation properties of powders. In more recent years, dry coating has emerged as a more efficient, sustainable and cost-effective method to modify powder surfaces. Wet coating, as well as other previously discussed particle size enlargement processes, requires large energy and material input in various process steps such as binder atomisation, powder fluidisation and powder drying and cooling, whereas the dry coating process produces no organic waste streams and also has the potential

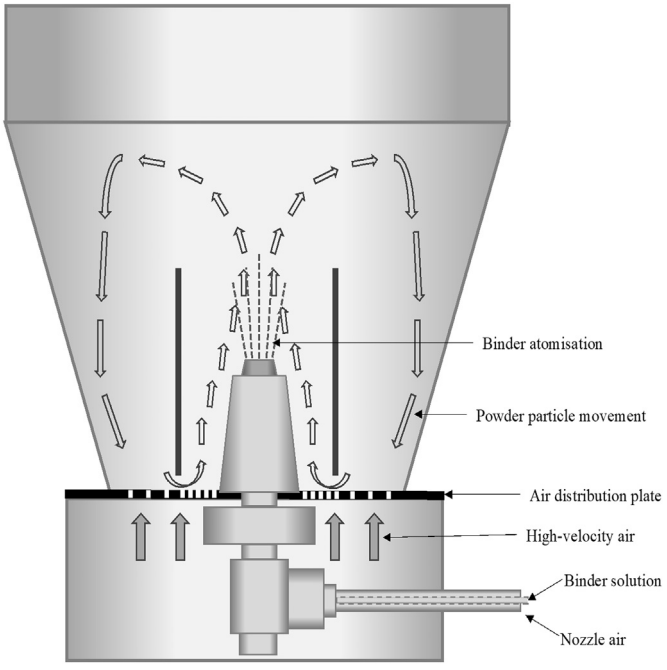


Fig. 7. Schematic representation of a fluid bed coating process achieved using a Wurster coater insert.

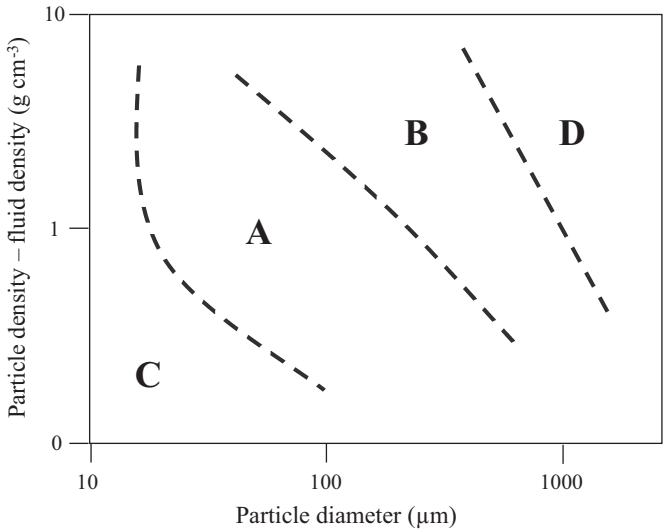


Fig. 8. Geldart classification chart for particle fluidisation: C- Cohesive, A- Aeratable, B- Sand-like and D-Spoutable.

to minimise energy input as no binder addition or drying steps are needed in this process. A reduction in energy and material input, in addition to minimising waste streams during processing is of increasing importance to production industries as continuous improvements in environmental sustainability are desired [118,119].

Dry coating is achieved by introducing very fine, submicron-sized material, termed "guest particles" into contact with larger micron-sized powder particles, termed "host particles". Through mixing and introduction of mechanical force, these guest particles (also termed "flow aids", "glidants" or "lubricants") will be dispersed, to some extent, on the surfaces of the much larger host powder particles. Due to the large differential in size, as well as the close arrangement between the host and guest particles, interparticle interactions will occur which lock the guest particles to the surfaces of the host powder particles. These particles stay locked onto the surface of the host as the force of attraction between the two particles is much greater than the force of gravity acting downwards on the submicron-sized guest particles [32,120–122].

A review of previously published work shows that three main materials are used routinely as the guest particles for dry coating processing, these being, silica, silicate and stearate particles [123–125]. The importance of the size of the guest particle was illustrated in a study by Yang et al. [124], where silica particles of six different sizes (ranging from 20 nm to 2 μm) where introduced as guest particles in the dry coating of cohesive cornstarch particles (15 μm particle size). The results of this study showed that the improvement in flowability of the cornstarch was inversely proportional to the guest particle (silica) size, i.e., the smaller the particle size of the guest particle, the more effective it was for dry coating applications. The addition rate of the guest to the host particles is also noted as a key process parameter in dry coating. An increase in the flowability of the bulk host powder is experienced on increasing levels of guest particle addition. However, a critical point is reached where further increasing guest particle addition level will lead to a reduction in powder flowability. This critical level varies dependent on the size of the guest and host particles but, for most studied powders and host particles, an optimal addition level of guest particles is typically 0.5–1.5% of the weight of the host powder bulk. At this optimal addition rate, full coverage of the host particles is typically achieved which improves the powders flowability. On exceeding this addition level, excess host particle build-up may occur at the powder particle surface which could lead to further interparticle interaction between neighbouring particles that may act to increase the cohesiveness of the powder [19,117,126].

Due to the very small particle size of the guest particles (nm range), they have a very strong tendency to self-aggregate via van der Waals interactions. Therefore, when the guest particles are added to the host, the mixing step that acts to disperse the particles to the host's surfaces is a critically important step, which can limit the quality of particle coating that is achieved. Traditionally, depending on the scale, manual mixing or dry blending (v-shaped blender) have been used to disperse guest particles onto the surfaces of the host, although results for these methods have shown incomplete partial particle coating (porous coating) due to self-interactions of guest particles. Recently, high energy dispersion techniques have been used to achieve complete coatings (continuous coating) on the host particles and these include magnetic-assisted impaction coating (MAIC). In MAIC a coil structure creates an oscillating magnetic field that mixes and causes the fluidisation of host and guest particles which encourages particle-particle and particle-wall collisions leading to a uniform dispersion of guest particles at the surface of the host powder. Although mechanical force is used to disperse particles in MAIC, it is classified as a "soft" coating process when compared to other high mechanical force dispersion techniques such as hybridizer coating which exposes particles to very large mechanical forces in order to achieve uniform coating. Although high mechanical force dispersion methods achieve very uniform and complete particle coatings, their application is limited for particles that are susceptible to breakage, such as high-protein dairy powders [32,118,124,127].

The flowability of very cohesive powders such as cornstarch (angle of repose (AOR) before dry coating: 50°, AOR after dry coating: 27°), ibuprofen (AOR before dry coating: 57°, AOR after dry coating: 48°), and lactose monohydrate (AOR before dry coating: 65°, AOR after dry coating: 38°) have been improved via dry coating in numerous studies [120,123–125,128]. Combining this with a reduction in waste streams and energy consumption, dry coating represents a potentially suitable method for the improvement of high-protein dairy powders post-spray drying.

4. Emerging technologies for improving the flowability of high-protein powders

All approaches to improve powder flowability presented herein have been utilised, to some extent at industrial scale. Continually, novel technological approaches emerge for various applications that may be utilised and applied to achieve an improvement in powder flowability. The following section will highlight some emerging technologies studied mainly outside of the field of dairy technology that may offer potential to improve the flowability of high-protein dairy powders.

4.1. Steam jet agglomeration

Steam-jet agglomeration is a form of particle size enlargement involving rewet processing. In fluid bed and shear agglomeration-based processes, viscous binding solutions are atomised onto the surface of powder particles, whereas in steam-jet agglomeration, an aerosol jet of high temperature (greater than 100 °C) steam is directed at free falling powder particles. As the steam-jet hits the powder particles, rapid condensation of the steam occurs, due to the large temperature difference between the particle and the steam jet. This condensation process forms water vapor at the surface of the primary powder particles which partially solubilises the components on the surface of the powder particles resulting in an increase in the surface viscosity and stickiness. The free-falling particles, now with increased surface viscosity, will adhere to each other on collisions via liquid bridging, forming an agglomerated structure which is then stabilised by drying the liquid bridges [129]. Person et al. [130,131] have shown that for skim milk powder (SMP), steam-jet agglomeration forms agglomerates of increased size (>400 μm) that also have higher mechanical integrity, and reduced breakage on handling and transport while still possessing an instant

nature (wetted in <30 s) comparable to industrially produced agglomerated SMP (i.e., using fines return). It must be noted, however, that a low agglomerate yield (19–33% agglomerates) was reported in these lab-scale studies with all other material produced being too small (<400 μm) or too large (>4 mm).

The clear advantage of steam-jet agglomeration, over the previously discussed particle size enlargement methods, is the use of steam instead of a viscous binding solution to achieve an increase in particle size. This minimises changes to the composition of the powder being processed, which is desirable in the production of high-protein dairy powders. However, this process is reliant on the efficient condensation of water vapor at the surface followed by the subsequent imbibing of the water droplets into the powder particle surface leading to an increase in surface viscosity. Contact angle measurements efficiently characterise the initial contact and imbibing of a water droplet at a powder surface, with a low contact angle indicating good powder wetting properties. O'Sullivan et al. [38] measured the contact angles of both SMP and MPI, with SMP (highly agglomerated using steam-jet agglomeration) having a low initial contact angle measurement (approx. 40°). The high-protein MPI powder showed a much higher contact angle (approx. 120°) indicating its poor surface wetting properties which may act to restrict the effectiveness of steam-jet agglomeration for high-protein dairy powders.

4.2. Static elimination

The build-up of charge on the surface of powder particles during conveying and further treatments (such as milling) acts to reduce the flowability of high-protein dairy powders through the occurrence of electrostatic interactions between oppositely charged powder particles (Section 2.4.2). Due to the danger of excessive charging of powders, and their contribution to dust explosions, research has been conducted to develop methods to dissipate charge from the surface of powder particles.

Static eliminators have been developed in nozzle form to reduce the surface charge of powders. These nozzles are constructed using ionizing needle electrodes, a power supply and a compressed air source, and generate ions, causing the surrounding environmental air to become ionised before the compressed air forces the ionised air away from the nozzle and into contact with a powder surface. When in contact with the charges present on a powder surface, the ions (formed by the static eliminator) will neutralise the surface of the powder particles. Multiple nozzle static eliminator configurators have been implemented and studied in powder conveying lines and storage silos in order to avoid excess charge build up due to safety concerns [132–134]. A study by Pingali et al. [135] has identified, and focused on the use of a static eliminator (and a slowly rotating drum) for the improvement of powder flowability. A series of pharmaceutical blends were initially tumbled in a rotating drum before the introduction of a static eliminator which acted to dissipate the charge on the powder particle surfaces within the drum. The effect on the flowability of the powder was analysed by measuring the expansion of the powder bed within the drum (via digital image analysis), which is linearly related ($R^2 = 0.979$) to the powder's flow index, i.e., powder flowability. Results from this study showed an increase in the flowability of all pharmaceutical powder blends tested due to the reduction in particle charge (e.g., the flow index of a milled pharmaceutical grade lactose powder increased by 41% on use of the static elimination process).

5. Conclusion and outlook

To ensure the forecasted growth of high-protein dairy ingredient powders is realised, actions must be taken to allow for improved tailoring of their physical and bulk properties. Currently, challenges associated with storage, handling and application of these powders, caused largely by their poor flowability, are acting to limit the commercial growth of these ingredients. The flowability of high-protein dairy powders is limited mainly by the occurrence of van der Waals and electrostatic interparticle interactions and therefore, an intervention which acts to reduce the occurrence and strength of these interactions is needed to improve their flowability. The current industry standard for improving the flowability of dairy powders is via agglomeration during spray and fluid bed drying steps of their production. Although many studies have clearly illustrated the improvement in flowability of high-protein dairy powders using this approach, undesirable consequences are also experienced, such as decreased powder bulk density and decreased particle dispersibility on rehydration (in particular for casein-dominant powders). Several of the alternative approaches discussed in this review are actively used in other industries (e.g., pharmaceutical and biochemical) to improve powder flowability and offer potential to the food industry to allow for greater ability to tailor and modify the particle, bulk and functional properties of future generations of high-protein dairy powders.

Declaration of Competing Interest

none.

Acknowledgements

The authors would like to acknowledge the Dairy Processing Technology Centre (DPTC), an Enterprise Ireland initiative, for financial support and permission to publish this work (Grant Number TC/2014/0016).

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