Chapter

Powder Technology

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Abstract

Combining two or more granular or powder ingredients requires a suitable mixing process, which can be either free or random flow with no attraction forces between the particles or interactive or orderly with the presence of large active particles that attract others forming stable clumps. Food systems have very complex properties that make it difficult to standardize the mixing process. In order to achieve an efficient mixture, diffusive and convective mechanisms must be combined, and its success is achieved with a predominance of homogenization over segregation. Powder products are typically used in industry as dispersion in a liquid and should have some properties such as good wettability, water incorporation, flowability, and instantization. To work with powder products, it is necessary to make determinations such as density, particle size, texture, and compaction force, among others. All these physical properties affect and determine the behavior of powdered products during storage, handling, and processing.

Keywords: physical properties, powder products, solid particles, mix process

1. Introduction

Power mixing involves several steps. The first one could be mentioned as a classification of the powder particles. Flowability can be the result of a good classification step. Consistent feed from bulk storage containers into feed mechanisms of subsequent processing operations is necessary. Interparticle forces, including van der Waals forces, capillary, and electrostatic forces influence the behavior of powder flow systems, as well as a small amount of moisture. Flow properties as angle of internal friction related to cohesion force of solids are also determined.

Powder mixing requires a statistical methodology before choosing the right equipment. Only in this way is a satisfactory result obtained, with the distribution of the mixture components as close as possible to ideality.

Segregation tends to occur whenever bulk material moves, and it occurs where differential forces act on different fractions of the mass of bulk material, and when differences in particulate properties cause a preferential movement of particles.

The quality of mix and characterizing it requires taking several samples and analyzing them, as a random way. Measurement of the mixing profile in real time with near infrared (NIR) provides the opportunity to study the dynamics of powder mixing and enabling a more comprehensive statistical analysis [1].

This chapter aims to present some aspects of this powder technology.

2. Sieving

Before starting a powder mixing process, a classification and maybe separation of the particles is necessary.

Sieving process is the separation of a mixture of grains of different sizes in two or more plots, through a sieving surface, which acts as a gauge that allows and does not let the grains pass. The final plots consist of more uniformly sized grain than the original blend. Mesh is the number of apertures of a screen of the same dimension in each linear inch, counted from the center of any wire to a point exactly 1 in (25.4 mm), or by a specified aperture in inches or millimeters, which is understood to be the free opening or space between the wires. Example: A granular material (−10 + 100) means that everything passes through a 10-mesh sieve (particles smaller than 1.68 mm) and nothing passes through a 100 mesh (particles larger than 0.149 mm). Screen opening is the minimum clear space between the edges of the openings on the sieving surface, given in inches or mm.

Particle size distribution is the relative percentage by weight of the grains that constitute the different size fractions present in the sample. It is one of the most important factors in evaluating the screening operation and is best determined by a full-size analysis using test sieves.

3. Powder mix

Mixture can be defined as the result of combining two or more ingredients. It can be granular or powdery. For such granular or powder mixtures to be formed a suitable mixing process is required. According to Pernenkil and Cooney [2], powder mixing is a crucial unit operation in the food industry.

Mixing is considered as a critical factor, especially in case of strong drugs and low dose drugs where high amounts of adjuvants are added.

There are two types of mixtures, non-interactive or random, and interactive or ordered. The first are those of free flow, being mixtures of uniform particle size powders or grains, without intraparticle forces of attraction, thus flowing with little interruption. Consequently, each different particle will have the same probability of being found in any portion of the mixture. Interactive mixing is formed when large active surface particles exist where other particles are attracted. They form stable clusters and the force between the particles belongs to different chemical classes [3].

According to Fellows [4], it is not possible to obtain a completely uniform mixture of powder products or particulate solids, but according to Singh and Heldman [5], the most important fact in a mixture is the reduction of random mix variation.

There are basically three mechanisms in mixing solids: diffusion, convection and shear. Shear can be considered as convective, and efficient mixing must be combined by diffusive and convective mechanisms. A purely diffusion process generates high efficiency in the mixing of individual particles, however, occurs at a low rate. The basically convective process is fast but less effective, exhibiting an ineffective final blend. For solids the diffusive mixture will only occur by mechanical agitation. The particles will change their collective or individual relative positions, and segregation of the particles may also occur, occurring when particles of different sizes, shapes or densities are mixed. A good mix occurs when there is homogeneity of the particles.

It is difficult to define and evaluate the powder mix; but certain quantitative measurements in solids can help estimate mixer performance. The proof of the mixer in practice comes from the properties it provides to the final blend produced by it (**Figure 1**).

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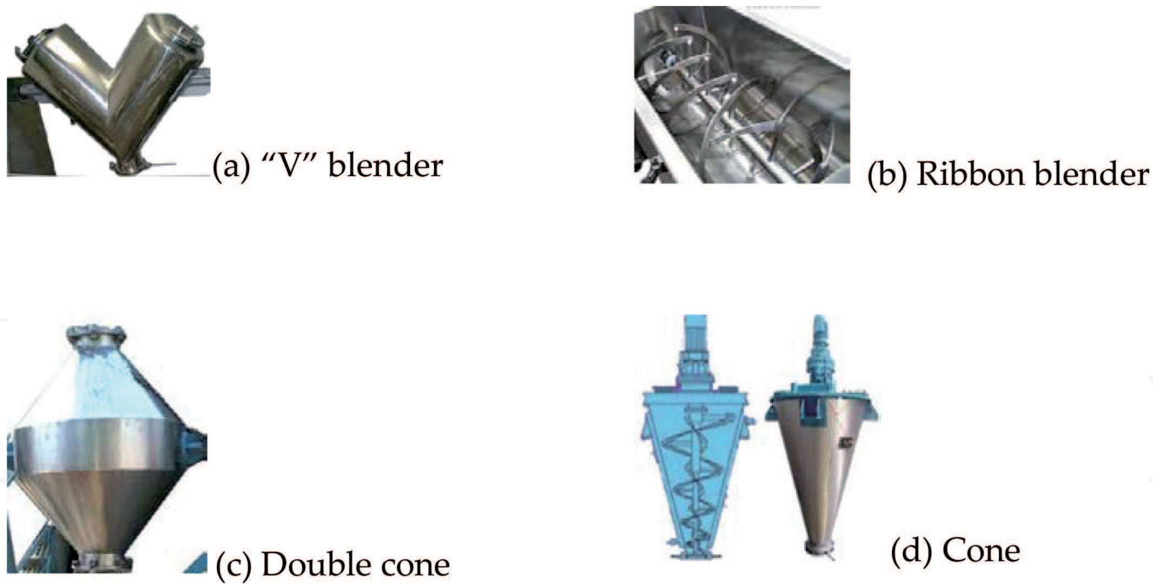

Figure 1.
Mixers.

The design of the mixer and its operation must be carefully chosen to achieve the desired results [4], as this influences the final product quality [6].

Mixing index involves the comparison of standard deviation of sample of a mixture under study with the estimated standard deviation of a completely random mixture (Eq. 1).

$$M_1 = \frac{\sigma_m - \sigma_\infty}{\sigma_0 - \sigma_\infty} \quad \sigma_0 - \sqrt{[V_1(1 - V_1)]} \tag{1}$$

where σ_∞ = the standard deviation of a 'perfectly mixed' sample, σ_o = the standard deviation of a sample at the start of mixing and σ_m = the standard deviation of a sample.

V = the average fractional volume or mass of a component in the mixture [4].

Due to the complexity of the properties of food systems, which may vary during the mixing process, it is extremely difficult to generalize or standardize the mixing operation for various new or traditional applications. The development of mathematical modeling for the food mixing process is also scarce, and it is necessary to consult established procedures for equipment design or scaling up [3].

Near-Infrared (NIR) spectroscopy can be used of in-situ as the basis for an in-line control system to optimize mixing time of food powder blends [7].

Some recommendations before starting the process: Determine which particle properties are required to solve your problem; For the form it is necessary to use an image analyzer. If not, assume that the measured size is an equivalent spherical diameter; for many non-spherical particles do not try different techniques for size checking. Many particles are globular enough to be considered spherical in order to do a job.

4. Physical properties

Powder products have different physical properties that must be measured and studied to obtain a product with the desired characteristics.

Detailed information on the physical properties of powder products is required, especially as they are complex products [8].

Some forces acting on the particles, as Van der Waals, electrostatic, and surfaces forces. Cohesive forces and frictional forces result in surface-surface interactions which resist the movement of particles, and they should be minimal. During mixing, the particles develop surface charge, which produces repulsions between particles, occurring random mixing, depending on surface properties, polarity, charge, and moisture.

Normally, powder products are used in industry as dispersion in a liquid. The wettability test is a simple test used by industry that provides the time parameter required for the powder to be absorbed by a liquid. Although maximum product wetting time is an arbitrary choice, powders in which 90% of the sample has already been dipped within 5 minutes are good wetting [9].

Powder flowability is defined as the ease with which a powder will flow under a specified set of conditions. Some of these conditions include the pressure on the powder, the humidity of the air around the powder and the equipment the powder is flowing through or from. Quantify powder flow characteristics are Compaction, Cohesion, Compressibility and Bulk Density. Flowability cannot showed as a single value or index, due to the combinations of physical properties of materials, the used equipment and processing.

Some physical properties of the powders such as angle of repose or rest angle are of importance for information on product flowability. During powder reconstitution, surface moisturizing water molecules tend to reduce inter-particle cohesiveness, thus allowing faster water penetration, so powders with high angle of repose have greater difficulty in incorporating water [10]. Powders with an angle of repose of up to 40° usually flow easily, if the angle exceeds 50° the flowability may be impaired indicating lower flowability. Particulate solids with up to 35° angle of repose have good flowability, those of 35 - 45° have poor cohesiveness, those of 45 - 55° have good cohesiveness and those above 55° are very cohesive, with low cohesive cocoa powder (45°) and cohesive (52°) cupuassu powder, for example [11]. Some powders show changes in fluidity with storage time [12].

The settled density of powders can be easily determined with a graduated cylinder (20 g sample) with some stirring to constant volume [12]. For example, cupuassu powder has 0.53 g/mL and cocoa powder 0.51 g/mL [11]. Shittu and Lawal [10] analyzed commercial chocolate and found values ranging from 0.49 to 0.81 g/cm^3. Eduardo [13] found values ranging from 0.28 to 0.94 g/cm^3 for chocolate drink powder from market.

Some powder properties:

1. Wettability

Time required for a specific amount of dust to be completely wetted when it is placed in water at a specific temperature. It is mainly related to particle size and shape, temperature and liquid type. Particle surface characteristics and fat content and characteristics if present and the correlation between wettability and fat content are inverse. Important analysis for powder products that will undergo the reconstitution process, as from the wettability analysis can obtain information about the product, such as its dispersibility and tendency to agglomerate formation. The wettability test is used by industry and it is the most important step in the process of reconstituting powder products.

Within this physical property of powders, there are some forms of measurement such as immersion, capillary rise, condensation and spreading. Immersion is the traditional method, which is used in powders that wet reasonably well [14].

2. Solubility index

Determines the ability of the powder to dissolve in water. It is defined as the volume of sediment in mL after centrifugation. The powder is dissolved in water at a certain temperature and centrifuged. The supernatant is removed and replaced with water and centrifuged before reading the volume of insoluble residue.

3. Bulk/tapped density

It is the weight of the powder divided by the volume occupied, usually expressed in g/mL. The sample is placed in an aluminum cylinder, heavy and beaten (100 or 1250 times).

4. Particle size distribution

Particle size is a determining parameter in the effectiveness of homogeneity in a powder mix when these are mixtures of two or more components of different particle sizes [15].

Sieves can be used. The dust sample is divided into fractions with different particle sizes by sieving, or by Laiser (Mastersizer Malvern Equipment).

Particle size distribution and particle size are of utmost importance when studying powder products. The particle size distribution can be represented graphically by the accumulated relative frequency (usually given as a percentage) or by size frequency histograms at certain intervals. It should be considered in the analysis that more than 20% of the material cannot be retained in the first sieve or bottom, and more than 30% of the material cannot be retained in any intermediate sieve [16].

The physical properties of powdered products affect their behavior during storage, handling and processing. Therefore, the determination of such parameters is of great importance for industries that use powders as raw material or even as final product. This is the case in the building materials, ceramics, pharmaceuticals and food industries, among others [17].

Properties of the ingredients of a match that affect the mixing of solids:

1. Particle size distribution—reports the material fractions at different size ranges.

2. Bulk density—weight per unit volume of solid particles. It is not a constant. It can be diminished by aeration and increased by vibration or mechanical compaction.

3. Particle shape—ovoids, blocks, spheres, flakes, chips, rods, filaments, crystals, irregular shapes.

4. Surface characteristics - surface area and the tendency to retain electrical charge.

5. Flow characteristics—rest angle and flowability. They are measurable characteristics determined in standardized assays. A higher rest angle indicates lower flowability. An object resting on an inclined plane begins to slide when the inclination angle is increased sufficiently to overlap the frictional force between the object and the plane. In general, if the angle exceeds 50° the powder will not flow satisfactorily. With about 25° will flow easily (**Figure 2**).

6. Reliability—is the tendency of the material to break during the handling operation. One should also consider the abrasion between the ingredients.

7. Agglomeration state—refers to the independent existence of particles or their adherence to each other, forming aggregates. The type and amount of energy employed during mixing and the friability of the agglomerates will influence aggregate breakdown and particle dispersion.

8. Moisture or liquid content in the solid—often a small amount of liquid is added to the solid to reduce dust or satisfy a special need.

9. Viscosity and surface tension—at the operating temperature of any added liquid.

10. Thermal Limitations of Ingredients—Any effect caused by temperature change must be observed.

Segregation mechanisms can occur with poor flow properties, particle size difference, difference in mobilities and in particle density and shape, transporting methods, dusting stage. Can be summarized as:

1. Fine particle percolation. If a particle mass is disturbed such that individual particles move, a rearrangement of the particle packing occurs;

2. Increase of coarse particles in vibration.

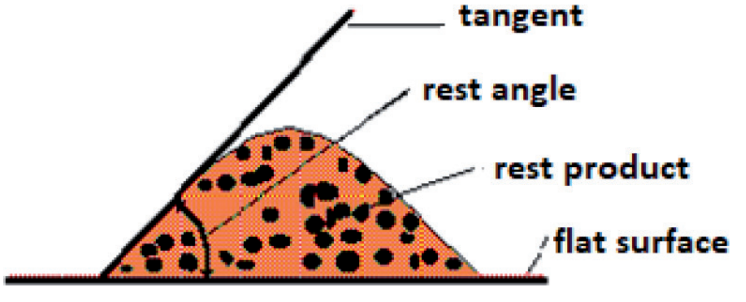

Figure 2.
Rest angle determination.

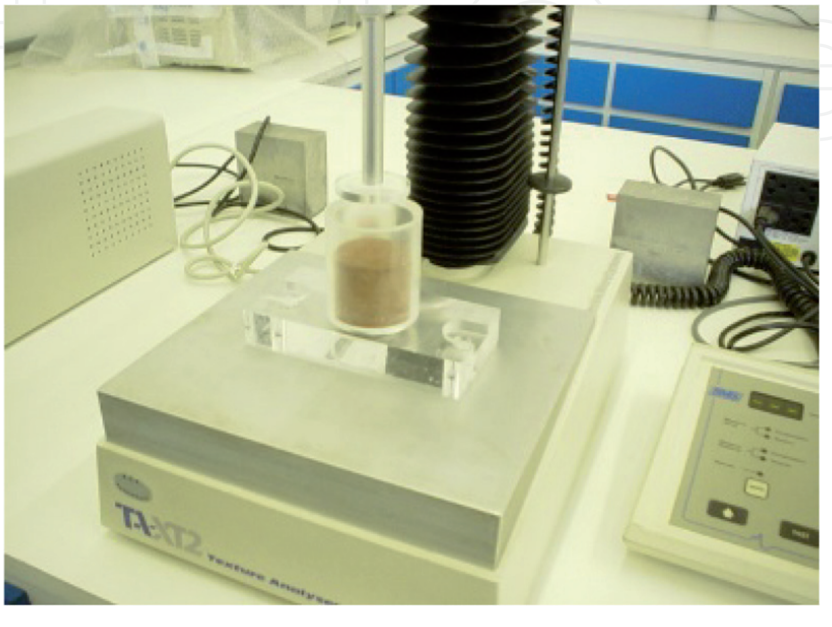

Figure 3.
Texturometer, back extrusion *probe and cylindrical cup with sample.*

4.1 Compression

Compaction can be understood as the compression of a two-phase system, solid and gas (dust and air), under the action of a force, which results in a reduction in the volume of the product. Compaction determination is useful for flow evaluation, friction tendency and dust agglomeration. In industry, the compaction process is used when forming powders, such as tablets. Under a compressive force, the particles rearrange (increasing the density of the dust), deform, and fragment [11, 13, 18].

However, these transformations continue to happen even when compression is not desired. Cartwright [19] associated the dispersibility of powders with their texture. He stated that very fine particles should be avoided when a good instantaneous powder is desired.

Eduardo and Lannes [16] developed a methodology for determining the compaction strength of powders using the TA-XT2 texturometer and the *back extrusion* probe. Medeiros [9] complemented with the compression distance test, aiming to determine the maximum volume reduction occupied by the sample, but that would not exceed the 20,000 g force. The compression strength test is performed at the distances determined in the first test (**Figure 3**).

The relationship between the compaction force and the compaction capacity of the sample is inversely proportional; hence, a sample is most compactable if its compaction force is lower. Based on these data, the most compactable sample was cocoa, as it presented the lowest compaction force, and the least compactable was cupuassu powder, there was no significant difference between them [16].

Eduardo and Lannes [13, 16] determined the compaction force of commercial chocolate, the results obtained ranged from 532 to 16,399 g. From these results the chocolate products were classified as very compact, with force below 2000 g and little compact, with force above 2000 g. These results also depend on the intrinsic characteristics of the particles, such as shape, size and homogeneity.

In granular materials (such as powders) pressure can cause permanent volume change. The removal of air between particles, causing a change in dust volume, can be caused throughout the storage period, transportation or even processing if some type of vibration is involved. Powder products contain in their formulation a great diversity of ingredients with distinct particle characteristics, and the reduction in volume is due to the accommodation of smaller particles between the space left by the larger ones (particle percolation).

5. Instantization

The instantization property identifies foods that are easy to solubilize in cold water, obtained in the drying process using dispersing substances, or through the action of agglomeration [9, 20].

Several physical and chemical methods have been employed to improve the instant properties of powdered foods, as is the case of adding cereal alcohol with its subsequent evaporation under controlled time, temperature and relative humidity, as showed by Barros [21].

One of the methods used to achieve instantization of powdered products is the spray-drying procedure that atomizes a solution by hot air [9, 22].

Spray-drying technology is widely used in various industrial segments including pharmaceutical and food. Although it is a technology that requires large investments in facilities and operation, there are many reasons why it is widely used. These advantages include consistent quality particle production, continuous use, the applicability of the technique to thermosensitive and heat resistant materials, the ability to process various types of raw materials, and the flexibility to define a project based in the formulation. To make use of these advantages, there are several aspects that must be considered. These include the evaluation of the formulation and process parameters, the specific type of particle to be produced and the properties of the material used [9, 23].

Mist drying is the transformation of low or high viscosity liquids, even those that are almost pasty, into dry and pulverized product in a single operation. The liquid or paste is atomized using a centrifugal or high-pressure system where the atomized droplets immediately meet a hot air flow. The rapid evaporation allows keeping the temperature of the product low. Heat and mass transfer are accomplished by direct contact between the hot gas and the dispersed droplets. Fine particles are separated from gas in external cyclones or collecting sleeves. When only the coarse fraction of the finished product is desired, the fines can be recovered in washers; washer liquid is concentrated and returned to the dryer [24–26].

The main use of spray dryers is the drying of solutions and aqueous suspensions. They are also used in combined drying and heat treatment operations. Feed is usually a liquid solution, suspension or paste that can be sprayed [9, 27]. The product to be dried goes through nozzles of varying sizes, influencing the particle size obtained, the liquid part is transformed into an atomized spray. The dust is carried in an airstream that carries it in contact with the spray.

Improvement of the physical and chemical characteristics of the materials used in this technique generally involves the comparison of process parameters such as heating, air volume, atomizer nozzle type, flow rate of the material to be dried or atomization system, drying air temperature. Formulation parameters are evaluated together with process parameters. It is important to check, for example, when the temperature is raised, if there is no extensive protein denaturation, loss of flavor, as well as impairment of solubility, stability and compaction [23, 28].

Heating and mass transfer during drying occur with air and vapor films around the droplets. This vapor shield keeps the particle at saturation temperature. As the particle does not become dry, evaporation continues, and the temperature of the solids does not approach the temperature of the drying outlet. Because of this, sensitive products can be dried at relatively high temperatures.

The shape of most atomized particles is spherical, which ensures fluid-like flow. This helps in the handling and filling process, for example, as well as in reducing costs. The particles still have homogeneity in the composition and the particle size distribution is very close, minimizing the obtaining of very fine particles, which is very important for the obtained product.

Factors such as humidity and water activity are of great importance in the study of the obtained product. Process definition and suitability of equipment operating parameters are particular to each desired finished product, depending on the characteristics it is intended to provide [29].

As the spray-dryer technique is widely used in the industry, the study of its potentiality and suitability to obtain powder products is a way to study the drying process for this and other products, as well as explore the equipment and its resources, obtaining a differentiated product. Instantization and improving product wettability are very important factors in obtaining a powder product, where the drying technique becomes a means to obtain these characteristics. Straatsma et al. [30] studied the solubility index of spray-dryer instantized materials, and this index is of primary importance for instantized powders. The thermal load of food products during drying is an important factor in the final quality of the powder, since heat exposure can lead to the formation of insoluble materials which are undesirable especially for instant powders. Spray-dryer equipment can be seen in **Figure 4**.

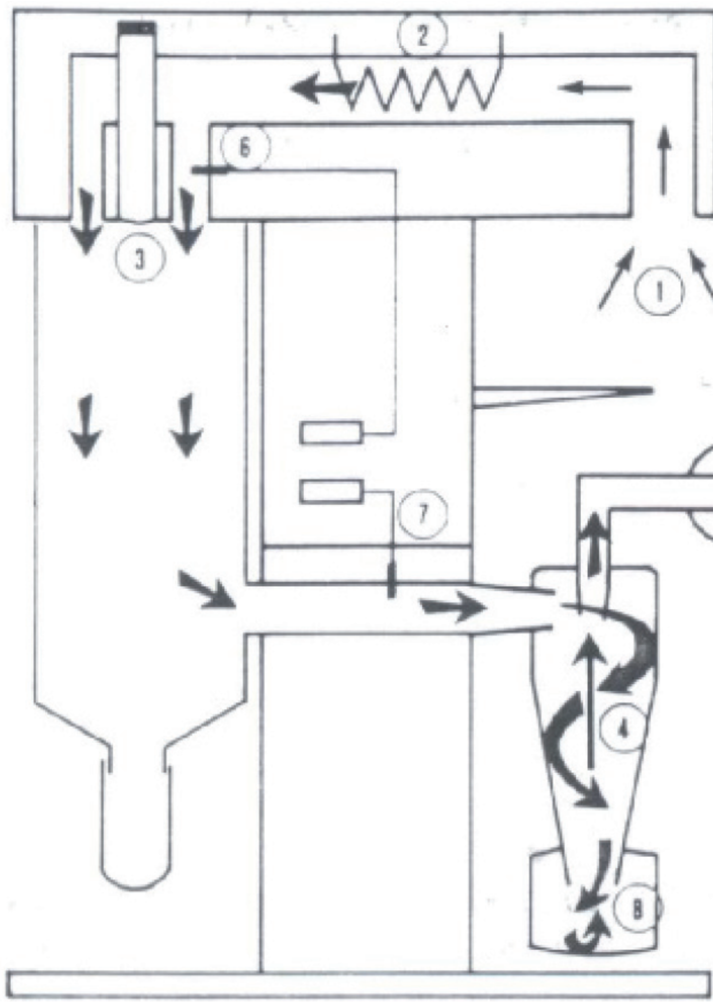

Figure 4.
Scheme of spray-dryer and drying air flow [31] 1. Air inlet, 2. Heating, 3. Entering the drying chamber, 4. Cyclone, 5. Vacuum Cleaner, 6. Control of inlet air temperature, 7. Control of outlet air temperature and 8. Receiving vessel of final product.

Optimal selection of inlet and outlet temperature differences is one of the most important aspects of spray-dryer. The outlet temperature cannot be chosen as desired as it results from the combination of inlet temperature - vacuum adjustment and product feed pump performance.

Product feeding and the introduction of the drying air in this type of dryer are performed at the top of the chamber in co-current flow system. Drying takes place while hot air and the product in the form of small droplets travel through the drying chamber to its conical base. Moist air and dry product then follow to the cyclone, where they are separated, and moist air is removed, and the dry powder product is collected at the base of the cyclone.

Atomizing a powder mixture involves a combination of ingredients, improving wettability in water or another liquid, evening out powder particles as well as improving their flowability and dispersibility. Its high cost must be offset by these factors [32].

Dispersibility is the ability of the powder concentrate to suspend in water to form finely divided particles that will remain in suspension for a reasonable period. It is described as a carrier surface feature and dispersing agents are added by overlapping the forces of attraction between the particles [33]. The dispersion of solids is affected by the texture of the powder, and to be instantaneous the powder must be optimal in size and very fine particles should be avoided [19]. Proper formulation requires a balance between aggregate size and interactions between different chemical additives, as well as adjustment of grinding process conditions.

The degree of atomization influences the drying rate, as well as the residence time of the particles influences the drying size. All atomization techniques can provide good control over the average particle size, but there are differences in their distribution [27].

The concentration of the input product in the atomizer influences the particle size, higher concentration of the solution, the more porous the particles obtained. The lowest concentration provides the smallest and finest particles. Higher flow of atomized product leads to smaller particles in the final product [19]. The adjustment of the process parameters, formulation, atomized product concentration, temperatures, spray speed, should aim at higher yield.

The quality of powdered foods is based on the properties variety that depend on specific applications. In general, final moisture content, solubility, rheological properties of the powder and density are of prime importance. Currently the main challenges in powder production are product development and process cost reduction. As a result, the production capacity is maximized, process conditions are directed to minimal product losses, reduced energy consumption, online quality control [29, 33].

Spray drying is nowadays a technology widely used in the food industry. The purpose is to protect thermosensitive active substances. Many researches are being developed using the microencapsulation method. Thus, to protect oils from lipid oxidation [34–36], incorporating functional ingredients such as vitamins [37, 38], additives and their storage protection [38], antioxidant protection [39].

6. Moisture sorption isotherms

Knowledge of sorption isotherms of powders blend is important for generating data for storage procedures such as shelf life prediction as well as drying processes when this is used in the process. Sorption isotherm can be defined as the graphical representation of the relationship between different humidity and water activity (parameter that describes the degree of binding of water to food particles) at constant temperature [40, 41].

In food, the microscopic structure is of primary importance in all aspects of its functionality. The microscopic organization of both water and other components determines the outcome of macroscopic observations made using different techniques [42–44].

Water activity of a product is defined as the ratio of water vapor pressure to pure water vapor pressure at the same temperature, and the availability of water-based criteria that can provide indicators of stability include water content, solute concentration and osmotic pressure.

7. Final considerations

A perfect mixture of two or more types of solid particles is one in which a sample contains the same proportion of components as any part of the mixture. Mixing of powders is a process that involves a comprehension of the physical elements of the mixture, equipment design, and appropriate sampling technique to ensure mix quality.

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