

## **Control of Ceramic Particle Properties by Spray Drying**

### **Introduction**

The use of spray drying in ceramic manufacture began in the mid 1950's for whitewares, e.g. floor and wall tiles. There followed a growth in electronic ceramics (ferrites, titanates, nitrides) and hard metals (carbides).

In recent years we have seen the emergence of advanced ceramics for new and varied applications including heat engines, turbines, high capacity batteries even for artificial bones, joints and heart valves.

Many of these novel applications have stringent requirements not just in terms of particle form but also in terms of purity cross-contamination and containment more commonly associated with the pharmaceutical industry.

The aim of this Paper is to outline the development of the spray process to meet these new challenges.

### **Process Overview**

Many products use the same basic manufacturing technique.

- Raw Materials
- Pre-Treatment
- Wet Milling
- Screening
- Drying
- Press
- Fire

This process may take place in an aqueous medium or in the case of non-oxides degrade by water then in the presence of a solvent.

It is normally a pre-requisite of the spray drying process that powder properties should be controlled with respect to:

- Bulk density.
- Particle size and distribution.
- Flow characteristics.
- Moisture Content.
- Compressibility.
- Shrinkage.

Pressing operations also require a specific press body quality to overcome product sticking in dies which leads to non uniformity in ceramic surfaces.

## **The Spray Drying Process**

The spray drying process comprises four steps, each of which influences the final product form.

- Feed preparation.
- Atomization/Hot air contact.
- Evaporation, particle shape formation and drying.
- Dried product separation from drying air and discharge.

## **Feed Preparation**

The first requirement is that the slip must be pumpable, homogenous, and free from impurities.

Typically the slip may contain some or all of the following:

- Organic Binder. e.g. PVA to hold the material together after pressing and prior to firing.
- Lubricants, e.g. glycerine or ethylene glycol to soften the particles, to aid pressing.

As the binders and lubricants are held in solution within the prepared feed each spray dried particle has an even coating of binder and lubricant upon its surface enabling binder concentration to be reduced to a maximum recommended concentration of 2% and lubricant to 1%. As these elements are burnt out in the kiln it is important to minimise their concentration in order to minimise shrinkage during the firing process.

- Defloculating agents allow the feed solids concentration to be maximised up to 85% solids in the case of stearite.

Increasing feed solids concentration not only improves the thermal efficiency and plant capacity of the spray drying operation but has a strong influence in the bulk density of the final powder.

Defloculants may be organic or electrolytic however care must be taken in the latter case as the salts within the water used to prepare the slip in combination with the electrolytic defloculant which remain in the press body may lead to powder sticking in the dye.

## Atomization

Choice of atomization method characterises the spray drying process.

Rotary atomization is typically used where there is a requirement for fine particles in the size range 50-150 micron or where the evaporative capacity exceeds 2 tonne/hr, whereas a pressure nozzle is the preferred choice where coarse particles in the range 200-300 micron are required.

For smaller dryers where the physical dimensions of the spray drying chamber make the use of a pressure nozzle impractical particularly where a particle size range 100 - 200 micron is required, then two fluid nozzle atomization is preferred.

## Rotary Atomization

The spray drying process using rotary atomization is characterised by a co-current drying regime, with an air disperser which imparts both horizontal and vertical vectors to the incoming drying air.



Chambers are generally of larger diameter than for nozzle atomization and must be oversized where larger particles are required.

Atomization occurs at the wheel periphery and it is the peripheral speed normally in the range 100 to 300 m/s which controls particle size for any given product.

One key element given the high abrasive nature of ceramics is the wheel design and the development of a fully abrasion resistant wheel with carbide bushings for such products.

The rotary atomizer has the advantage that high feed rates can be accommodated by a single atomizer, it requires only a low pressure feed system, it is resistant to abrasion or clogging and that particle properties are not sensitive to feed rate.

## Nozzle Atomization



### Pressure Nozzle

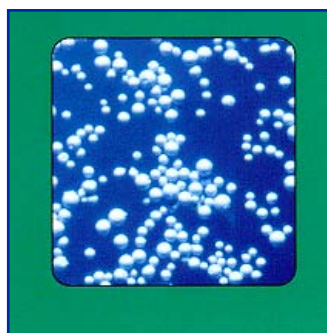
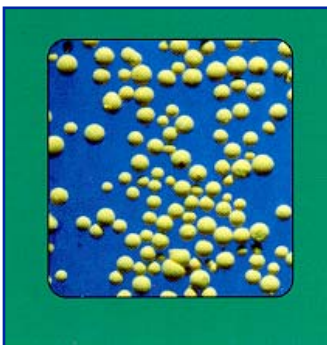
In the hydraulic pressure nozzle atomizer slip is supplied to the nozzle under pressure. The pressure energy is converted to kinetic energy and the feed issues from the orifice as a high speed film that readily disintegrates into droplets.

The droplet size produced from a pressure nozzle varies inversely with pressure and directly with feed rate and feed viscosity.

High capacities cannot be accomplished in a single nozzle and require multi-nozzle systems which may complicate start up operation and shut down procedures.

### Two Fluid Nozzle

The third alternative for atomization is the multi-fluid nozzle of which the two fluid nozzle is an example.

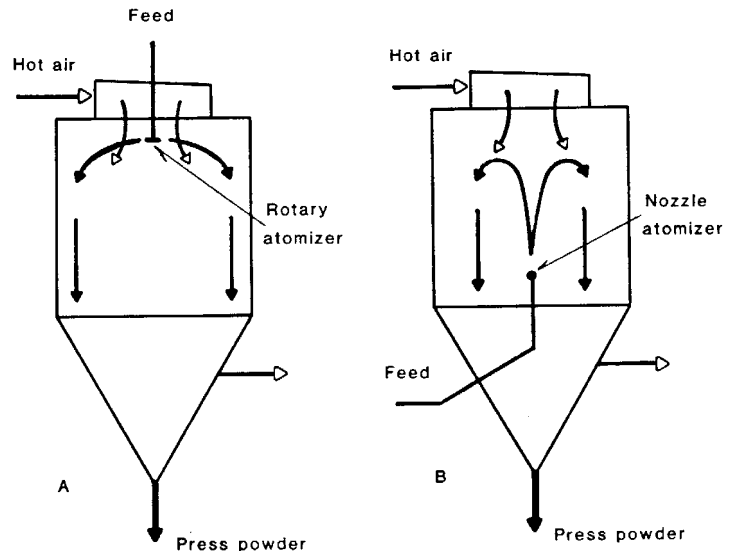


In this atomization device the energy for atomization is provided by the rapid expansion of gas which is mixed with the feed within the body of the nozzle (internally mixing) or at its tip (externally mixing).

Particle size is controlled by the feed to air ratio.

Whilst this type of atomization is not generally considered economic for high capacity applications, it is a suitable alternative for smaller specialist plant due to its relatively low pressure and resulting particle velocity and shorter required drying path.

Dryers utilising nozzle atomization are generally of a smaller diameter but with an increased cylindrical height. The increased particle trajectory achieved by the counter co-current or fountain nozzle configuration in combination with the streamline air flow allows production of coarser particles without wall deposits.



## Evaporation

Evaporation takes place from the saturated vapour film which is quickly established on the droplet surface.

Droplet surface temperatures remain low - approximately equal to the wet bulb temperature of the drying air. This is the so-called "constant falling rate condition".

The residence time of the particle within the chamber must be sufficient to allow total water removal.

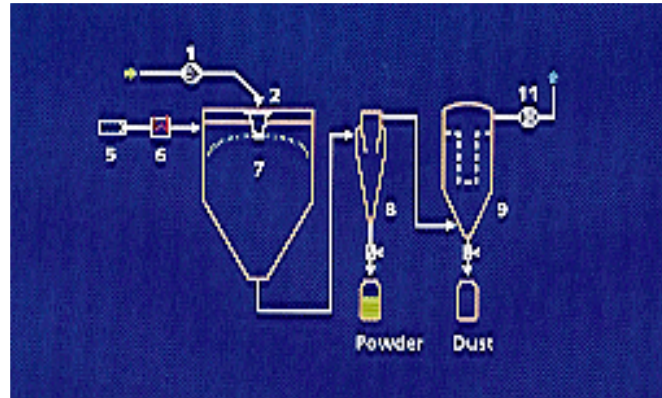
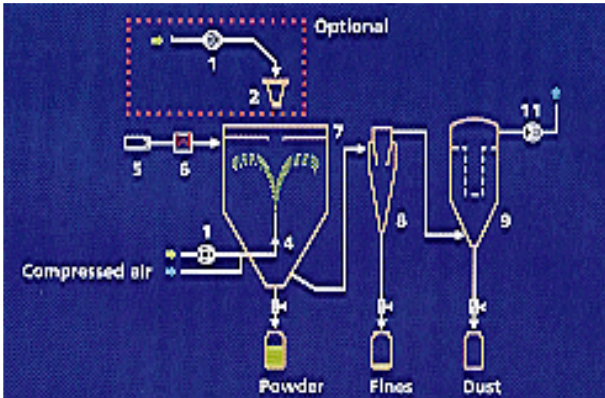
During the evaporator process the atomized spray size distribution undergoes change as droplets can shrink as moisture is evaporated, agglomerate or lose sphericity due to distortions during the initial drying phase.

In the case of rotary atomization this initial phase is influenced predominantly by the dryer outlet temperature and to a lesser extent by the inlet temperature whereas for a counter co-current design the thermal history of the particle is a factor of both inlet and outlet temperature.

## Separation

The separation of the particulates from the drying air can be achieved in one of two ways.

The coarser material may be collected from the cone of the spray drying chamber with the finer particles recovered from the drying air by a primary separation device - cyclone or bag filter. The two powder streams may be subsequently mixed or segregated, often the coarser chamber fraction being recovered as product, whilst the fine material is recycled due to its superior press characteristics.



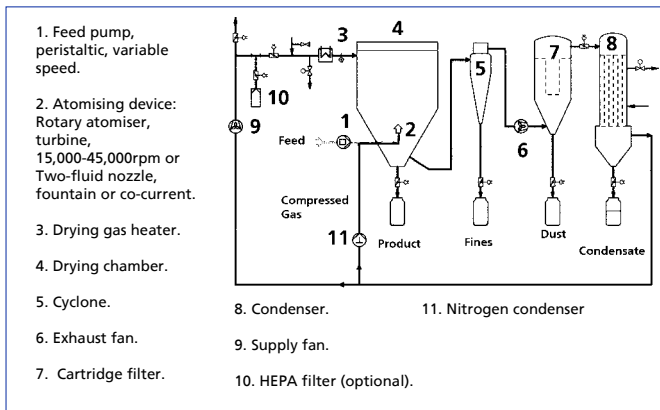
Alternatively all product may be collected from the separation device resulting in a wider particle size distribution and increased bulk density.

### New Generation of Advanced Ceramics

Over recent years we have seen the emergence of specialist ceramic materials, carbides, nitrides and composites for a variety of applications, typically of small batch size but requiring specific particle properties:

- Individual particles, either fine or coarse.
- Absence of misshapen particles and agglomerates.
- Controlled particle size distribution.
- Total absence of impurities or cross-contamination.

#### Closed Cycle System



These requirements have seen the development of a number of specialist plant designs sharing many of the features previously associated with the pharmaceutical industry.

Such plants may be designed to operate with air as the drying medium for the drying of aqueous products or under nitrogen where the slip is suspended within a solvent.

The spray drying of a non-aqueous feed may take place using a total loss nitrogen system with a carbon filter placed after the powder

separation device (bag filter) to absorb the solvent prior to the discharge of the drying gas to atmosphere or in a closed cycle loop where the absorber is replaced by a condenser and the drying gas is returned to the inlet of the process and recompressed for use in atomization in the case of the two fluid nozzle.

Such plants must be designed to ensure that the system is fully purged of air prior to commencing the drying operation, that it is gas tight to protect the operating personnel from exposure to the drying gas and that on completion of the drying operation the plant be fully air purged prior to any operation intervention, for instance, cleaning. This requires comprehensive and fail safe instrumentation both on the plant itself and within the operating environment.



Whether they be open or closed cycle, aqueous or non-aqueous the advanced ceramic plants must satisfy a number of criteria.

### **Absence of Contamination**

In many applications the presence of even minute traces of impurities cannot be tolerated. This as in the pharmaceutical industry has been addressed by:

- The incorporation of very high standards of air filtration of the drying air (HEPA filtration) immediately before its entry into the spray drying chamber.
- Where practical/necessary the installation of the dryer itself or the product discharge in a controlled clean room environment..
- The incorporation of effective Cleaning-In-Place (CIP) systems on the main dryer components with easily dismantled ductwork.
- Attention to surface finishes.



### **Control and Reproducibility**

In order to consistently produce an identical product all variables must be precisely controlled and monitored, which includes all aspects of feed preparation and drying conditions. This requires not only an enhanced level of instrumentation but that all instruments must be regularly checked and calibrated.

## **Conclusion**

Spray Drying as a technique allows the production of ceramic press powder with a wide range of product characteristics in a controlled and reproducible manner.

The more stringent requirements of hi-tec ceramics production offers new challenges, many in common with the food and pharmaceutical industries which call for great care to detail in the design operation and documentation of plant design.

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