Formulation of Highly Concentrated Suspensions for Spray Drying in a Fluidized Bed†

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Abstract

Spray drying of suspensions leads most often to free-flowing powder products of good quality. However, this process is energy-consuming, especially because of the high amounts of liquid to be evaporated from the sprayed suspension. Indeed, in order to ensure a good quality and stability of spraying, the amount of solvent must be determined so as to avoid excessive suspension viscosities. For a given amount of solvent, it is also possible to improve the viscosity of concentrated suspensions by deflocculation using appropriate dispersants.

Through the use of an adequate dispersant, we showed that it is possible to obtain fully dispersed suspensions and to optimize their dryness in order to reduce the moisture content to be evaporated. The effect of different salts and the molecular weight of polyacrylic acid is investigated to improve the formulation of concentrated calcite suspensions with the focus on spray drying.

A new continuous process, developed to enable the drying of highly concentrated slurries at low temperature, is also described. This process is based on the coupling of coating/attrition of a suspension in a fluidized bed of inert particles, and it strongly increases the surface area available for heat and mass transfers.

Further analysis of this process has been carried out addressing the drying kinetics in a drying pipe. It shows a strong influence of the support wettability as well as the surface tension of suspension on the drying kinetics of suspension films. The surface tension of the suspension and humidity were found to be crucial parameters to optimize the powder recovery.

Keywords: spray drying, inert particles, suspension, drying kinetics, attrition

1. Introduction

The drying of bulk materials plays an important role in a lot of industrial processes. Most of the processes of manufacturing mineral powders use a thermal dehydration of the initial suspension followed by a mechanical milling step.

The spray drying process can also be used, but it is a high-cost drying technology especially if the initial suspensions have a low solid content. Spray drying involves the evaporation of moisture from an atomised feed by mixing the spray and the drying medium – typically air. The drying proceeds until the desired moisture content is reached in the sprayed particles and the product is then separated from the air. Low viscosity is essential to the ease of pumping and to a homogeneous spray of the suspension (Tsetsekou et al., 2001).

Sometimes, mechanical filtration can be a partial alternative to the thermal dehydration process in order to obtain a concentrated suspension. This step can be followed by spray drying if the filtration cake can be pulverized. Unfortunately, it is not always possible because of the high viscosity of suspensions. New drying processes for these types of highly concentrated suspensions have to be developed to enable a lower production cost of the dried powder.

Moreover it is well known that in order to decrease the viscosity of concentrated suspensions and in order to improve their stability, it is necessary to avoid the agglomeration of particles and to enhance their
deflocculation. For this purpose, dispersants can be used as deflocculation agents. Several studies have shown the effect of dispersants on the rheological properties of slurries (Tari and Ferreira, 1998, Kugge and Daicic, 2004). Polyelectrolyte dispersants such as the salts of polyacrylic acid can be used to prepare concentrated suspensions of mineral powder (Garcia, 2001, Tobori and Amari, 2003) and can improve the rheological properties of concentrated calcite suspensions.

Recent work carried out in the chemical engineering department of the Compiègne University of Technology led to development of a new filtration process of mineral suspensions (Husson et al., 2002). This process uses dispersing agents in order to obtain a highly concentrated and easily fluidifying final cake (Mouroko-Mitoulou, 2002). With this process, the mechanical dehydration can lead to pulverizable concentrated suspensions which can be dried by thermal dehydration, e.g. atomization. Hence, this process is important from an energetic point of view.

Furthermore, fluidized beds are well known for their high heat transfer capacity. Conventional fluidized bed dryers are not efficient enough to dry concentrated slurries. Recent works have developed new techniques for slurry spray drying which ultimately enables low drying temperatures for temperature-sensitive products.

These emerging processes are based on the coating followed by the attrition of a suspension sprayed onto fluidized inert particles. The drying mechanism of this process relies on several concomitant steps as showed in Fig. 1. First, the sprayed suspension sticks to the heated particle surface. This deposited coating then dehydrates and peels off from the inert bodies because of the interparticle friction. The crushing of the coated layer occurs due to intensive interparticle collisions induced by the fluidized bed flow, allowing removal of the dried product by elutriation. The dried powder is then conveyed by the fluidizing air to a cyclone or a filter for collection (Grbavcic et al., 2004).

Various geometries and configurations for these types of processes have been developed and patented, for example by Zedlets and Bogdanov (1977), Lukach et al. (1978), Dobronogov et al. (1981), Shmaryan (1981), Chevikov (1981), Dvortsin et al. (1982), Kozachek and Shikunov (2003).

Several researchers have shown that the turbulent mixing induced by the flow of inert particles enables slurries to be dried in fluidized bed dryers containing inert particles such as sand (Mousa, 1979), glass spheres (Grbavcic et al., 1998) or Teflon cubes (Benali and Amazouz, 2006). These inert particles – used as heat conveyors – strongly increase the surface area available for heat and mass transfers.

More recently, Marques et al. (2006) studied the influence of the surface energy – of the inert particles – on the drying effectiveness. This study revealed that highly hydrophilic particles lead to a difficult recovery of the dried powder because the coating phenomenon becomes predominant compared to the peeling/drying mechanism. However, highly hydrophobic materials can prevent adequate coating of the liquid to be dried on the inert particles and consequently reduce heat transfers due to a smaller surface available for the transfers. The choice of materials also seems to depend on the surface tension of the liquid to be dried.

However, there is a lack of information in the literature regarding the understanding of these phenomena on a small scale to better understand the processes of drying in a fluidized bed of inert particles.
2. Experimental

2.1 Materials

Suspensions were manufactured by introducing dried powder into an aqueous solution of dispersant. Calcium carbonate, supplied by Verbiese (France), was chosen as the raw material for its high commercial interest. The median diameter of CaCO₃ particles was 3 µm with a density of 2.74 g/cm³. The size and morphology of the CaCO₃ used can be seen on the SEM photographs in Fig. 2. Previous researchers (Dusanter et al., 2007) showed that the optimized solid content of the suspension – leading to the best agreement between a high CaCO₃ content and sufficient sprayability on the apparatus – was 60% of CaCO₃ w/w. Dispersing agents were four aqueous solutions of polyacrylate salts. They differ by the type of their counter-ion (sodium or ammonium) and the medium molecular weight of polymers (approximately 2500 and 5000 g/mol). Characteristics of these dispersants are summarized in Table 1. The dispersant concentration was fixed as a percentage of the mass of active polymer per mass of dry calcium carbonate powder for all suspensions.

2.2 Suspension drying

Conventional spray drying tests were carried out using a BÜCHI mini spray dryer B-190 (Fig. 3). This dryer works in co-current flow, i.e. the sprayed product and preheated drying air stream flow in the same direction. The suspension is sprayed by means of a two-fluid nozzle. The particles are then separated by a cyclone. The finest particles are collected in a filter bag. For the experiments, the air inlet temperature was kept constant at 175°C.

The process of spray drying fluidized inert particles relies on the atomization of the highly concentrated suspension in a fluidized bed of inert particles. The experimental apparatus is shown in Fig. 4. It consists of a cylindrical column in which inert particles (glass spheres 630-800 µm) are fluidized. An electric heater located upstream of the fluidization column supplies the air at the desired temperature by the use of a PID regulator. The spraying system is a two-fluid nozzle located in the middle of the fluidized bed. The suspension is supplied by means of a piston pump and sprayed downwards within the fluidized bed of heated particles. In order to provide a vigorous fluidization, the excess air velocity (U-Umf) was fixed at 0.11 m.s⁻¹. This velocity ensured an appropriate fluidization of inert particles (Umf = 0.33 m.s⁻¹) and the elutriation of dried peeled particles. At the column outlet, the air passes through a box containing a filter cartridge where the particles are collected.

The inlet air temperature was fixed at 80°C in the wind box. As soon as the temperature in the fluidized bed became stable (71.5 ± 0.2°C), the suspension was sprayed within the fluidized bed (feeding flow: 5 ml/min). It could thus be observed that the glass spheres gradually became less opalescent (coating). This step is quickly followed by the appearance of white dust ascending to the top of the column due to abrasion of the coated layer. Due to the large surface area of the glass spheres and the fineness of the deposited coating layer by spraying, the drying occurred in a very short time. During the feeding and after a stabilization period, the bed temperature remained stable at 52 ± 3°C.

2.3 Analysis apparatus

Several methods were used to characterize initial suspensions and powders obtained by standard spray drying or the spray drying of fluidized inert particles.

A rotation coaxial rheometer VT 550 (HAAKE) was used to study the viscosity, η, of initial suspensions, by measuring the strain at different shear rates (γ = 0 to 500 s⁻¹, 500 s⁻¹, and 500 to 0 s⁻¹). The temperature was fixed at 20°C.

An environmental scanning electron microscope (Philips XL30 ESEM-FEG) was used to observe the

| Identification | Counter-ion | Medium molecular weight of polymers | Content of active ingredient |
|----------------|-------------|-------------------------------------|-----------------------------|
| PNH₄_5000      | Ammonium    | 5188 g/mol                          | 42.2                        |
| PNa_5000       | Sodium      | 5733 g/mol                          | 43.6                        |
| PNH₄_2500      | Ammonium    | 2545 g/mol                          | 38.8                        |
| PNa_2500       | Sodium      | 2551 g/mol                          | 43.3                        |
shape and size of the CaCO₃ particles.

A Blaine permeameter was used to estimate the fineness and the specific area of dried powder. The basic principle of this test consists in measurement of the time necessary for a given amount of the air to flow through a packed bed of powder of a known volume. The Blaine specific surface \( B \) (cm²/g) is thus calculated using the following equation:

\[
B = \frac{K}{\rho} \cdot \frac{\sqrt{e}}{1 - e} \cdot \frac{\sqrt{t}}{\sqrt{0.1 \cdot \eta}}
\]

where
- \( K \) is the Blaine apparatus constant,
- \( \rho \) is the porosity of the packed bed,
- \( t \) is the time taken by the fixed quantity of air to flow through the powder bed,
- \( \rho \) is the powder density, and
- \( \eta \) is the viscosity of air at the temperature of experiment.

2.4 Device for determination of the drying kinetics

In this section, the study of the drying kinetics of thin layers of calcium carbonate suspensions by convection on microscope glass plates is described. The experiments were carried out in a drying pipe where microscope glass plates coated with suspensions are positioned in parallel to the drying air flow (Fig. 5). The device is inspired by previous works of Camisard (1997). The drying kinetics were studied by applying a film of suspension onto microscope glass plates previously subjected or not to a hydrophobic treatment by silanisation.

Drying occurred by means of air coming from the system of compressed air. The air flow rate was
controlled using a mass flowmeter placed upstream of the heating zone. This air was pre-heated by a heating ring surrounding the drain in which the air circulates. The temperature of the drying air was controlled by a PID regulator based on a Pt100 probe placed downstream of the heating zone.

The drying pipe was made of a polymethyl methacrylate tube in which the sample to be dried was positioned on a sample carrier, itself positioned on a balance. The acquisition device recorded the temperature and relative humidity evolution of the inlet and outlet air as well as the mass variation of the sample.

The film of suspension was applied to the glass plate – heated to the temperature of the test beforehand – using a coating bar “K Hand Coater” enabling the application of a 100-µm-thick film.

2.5 Device for estimation of the adhesion/attrition mechanism

Shear tests (block shear) were carried out, taking the ASTM C-273 standard as a starting point. The assembly must enable a relative displacement of two surfaces (i.e. microscope glass plates) of the sample, the joint (suspension film) being then solicited parallel to the surfaces of joining (Bretton and Villoutreix, 2005). Either one – for simulating the adhesion between a coated glass bead and an uncoated one – or both – for simulating the sticking together of two coated glass beads – microscope plates were coated with suspension (see Fig. 6) using the same coating bar as previously (2.4). The sample was then dried over a period of at least 14 hours at 60°C and then conditioned at the temperature and relative humidity of the test room for at least half a day before testing.

The shear test was performed using an Instron 4464 compression machine with a 2 kN load cell (Fig. 7) chosen according to the range of the experimental measurements (from about 10 to 520 N). The compression test enabled the shear modulus of suspension films to be calculated by the following equation:

$$G = \frac{\sigma_c}{\varepsilon_c} = \frac{F_{\text{max}}}{S} \cdot \frac{E_{\text{dmax}}}{d_{\text{max}}}$$

with

$$S = l \cdot L \cdot \left(1 - \frac{S_{\text{uncoated}}}{S_{\text{total}}} \right)$$

where $S_{\text{uncoated}}$ stands for the uncoated part of the specimen – due to the introduction of air bubbles during the sticking step, $l$ and $L$ are the specimen length and width, respectively.
3. Results and Discussion

3.1 Characterization of initial calcium carbonate suspensions

It is important to control the rheological properties of suspensions so as to attain a high solid content with sufficient fluidity to be pulverized. Preliminary experiments (Dusanter et al., 2006) were carried out to assess the influence of shearing time on stress $\sigma$ of dispersed CaCO$_3$ suspensions containing 1% of dispersant (CaCO$_3$ content varied from 30 to 70%). The shear thinning behaviour indicated a linear behaviour up to a critical rate and was associated to a Bingham behaviour fluid, showing that a minimum stress is necessary to break the residual agglomerates in the suspension (Vorobiev et al., 2004).

By applying the Bingham model ($\sigma = \sigma_y + \eta_p \dot{\gamma}$) for each rheogram, it was then possible to study the effect of dispersant content on plastic viscosity for different counter-ions and different molecular weights of the dispersants used.

Fig. 8 and Fig. 9 show that plastic viscosity evolves in various ways as a function of the dispersant content in the suspension: - a first decreasing part for which the viscosity depends on the concentration of dispersant, highlighting the progressive deflocculation of calcium carbonate agglomerates, - a second zone - where the plastic viscosity remains stable - probably representative of the optimum concentration of dispersant, - a last part with a strong increase of the plastic viscosity of suspensions when the concentration of dispersant grows. This phenomenon probably corresponds to a destabilization of the suspension by reduction of the zone of interparticle depletion. Indeed the surplus molecules of dispersant which could not be adsorbed on the surface of the particles tend to become organized in the form of polymeric balls. These balls behave like additional particles and decrease the available space in the dispersing solution. Thus they reduce the effect of steric repulsion. If these forces become higher than the repulsion forces between the particles, they can induce a destabilization of the suspension promoting an increase of the plastic viscosity. So when the polymer concentration is higher than necessary to recover the particles, an aggregation by depletion is observed. In our case, the minimum concentration of dispersant to recover the totality of the calcium carbonate particles

![Fig. 7](image1.png)

Fig. 7 Experimental device for the estimation of the adhesion/attrition mechanism. (specimen before testing)

![Fig. 8](image2.png)

Fig. 8 Influence of the dispersant quantity and the type of polycrylate counter-ion on the plastic viscosity of calcium carbonate suspensions.
was about 1%.

The type of concentration – sodium or ammonium – (Fig. 8) seems to have no influence on the plastic viscosity of suspensions. This point is confirmed in some work by Geffroy et al. (1997), showing that only the anionic part – i.e. the polyacrylic part – plays a role in the dispersion of calcium carbonate particles.

Concerning the effect of the molecular weight of the polymer (Fig. 9), the viscosity of suspensions is slightly higher for the dispersant with the lower molecular weight. Geffroy et al. (2000) showed that for polymers with a low molecular weight, the fractions with a molar mass lower than 1500 g/mol are not adsorbed. We can thus suggest that the quantity of dispersant adsorbed is less important for the dispersant PNa_2500, which explains why the corresponding suspensions have a higher viscosity, since the agglomerates are less dispersed.

3.2 Powders obtained by suspension drying

In this work, we investigate the drying of the concentrated suspensions by two spray drying methods: the conventional one in a laboratory dryer and an emerging one which consists of spraying the suspension in a fluidized bed of glass spheres.

The parameters of drying for both processes are summarized in Table 2. One can first notice that the process of drying in a fluidized bed of inert particles makes it possible to significantly decrease the drying temperature.

| Table 2: Operational parameters of the drying processes |
|--------------------------------------------------------|
| **Inlet temperature** | **175°C** | **80°C** |
| **Inside temperature before pulverization** | 100 to 110°C | 71.3 to 71.7°C |
| **Drying temperature** | 90 to 95°C | 49 to 55°C |

1% dispersant and 60% CaCO_3 – is shown in Fig. 10 (a). First of all, essentially all the granules are rounded. The structure of such a granule at high magnification shows that each granule is composed of a particle agglomerate arrangement of the rhombohedral calcium carbonate particles which constituted the initial raw material – used to prepare the suspension – presented in Fig. 2.

An SEM image of the calcium carbonate powder obtained by spray drying on fluidized bed of inert particles is shown in Fig. 10 (b). The fragmented agglomerates observed on this figure are very different from the ones obtained by conventional spray drying. No spherical agglomerates were observed in the powder, which implies that the drying by coating is predominant with regard to the drying of CaCO_3 on fluidized inert particles.

Moreover as one can see, the particle size of the powder particles is close to that of the initial raw material (Fig. 2). This can be explained by the breaking of aggregates which were previously formed by the dried coating on the surface of glass beads. Indeed, fluidization of the glass spheres creates a lot of collisions and thus leads to attrition of the aggregates. As
a consequence, this drying process could avoid an ul-
terior grinding. Although the fineness of the product 
can lead to a worse flowability, this process remains 
more interesting from an energetic point of view be-
cause of better heat transfer coefficients.

• **Blaine surface area**

The morphological observation of powders is con-
firmed by a Blaine surface area of 12317 cm$^2$/g for 
the product dried on inert particles which is close to 
the raw material value (13470 cm$^2$/g), whereas the 
powder obtained by conventional spray drying has a 
specific surface of 5972 cm$^2$/g. However, by decreas-
ing the concentration of dispersant in the suspension 
pulverized by the laboratory spray dryer (i.e. 0.1% in-
stead of 1% w/w) or by lowering its solid content (i.e.
30% instead of 60% w/w), it is possible to increase the
specific surface. Indeed, in these cases, the granules 
formed during drying are more friable so the specific 
surface of the powder obtained increases. Thus one 
can obtain a Blaine surface area which is close to 
10000 cm$^2$/g – depending on the drying conditions –
but this value still remains lower than the one ob-
tained by the drying process in the fluidized bed of 
glass spheres. Moreover the process would be more
difficult to control or would require more energy to 
evaporate the solvent due to more diluted suspen-
sion.

### 3.3 Determination of the drying kinetics

Parameters which are studied in the drying pipe are 
the temperature and flow rate of the drying air, 
the initial dispersant content of the film, and the wet-
tability of the support.

Typical recorded curves of the tests ([Fig. 11](#)) 
present the variation of the mass sample (LOD, in g 
of loss on drying per g of solid) and the evolution of 
the inlet ($T_i$) and outlet ($T_o$) air temperature and rel-
ative humidity (RH and RHo in [Fig. 11](#)) versus time.

![Fig. 11](#) Typical experimental curves obtained in the drying pipe ($Q_{air} = 50$ L/h, $T=40^\circ C$)
related to the mass of calcium carbonate. The loss on drying in the suspension film results in a decrease of the drying air humidity after the sample. Taking into account the small quantities of evaporated water, the outlet temperature of the air does not vary.

It appears (see Fig. 12) that the increase of the drying temperature slightly increases the kinetic rate of dehydration, but also especially reduces the quantity of remaining water (which drops by 9% to 3% for an increase of 10°C).

Concerning the influence of the hydrophobicity level of the plate (Fig. 13), it seems that the use of a hydrophobic support reduces the kinetic rate of water evaporation. However, the film application on this kind of plate is more difficult than the application on untreated supports (hydrophilic) because of their poor wettability. In this case, the film thickness is thus less homogeneous and locally higher, which leads to a reduction in the thermal heat-transferring surface.

In the same way, in the absence of dispersant and for hydrophobic supports (Fig. 14), the quantity of remaining water is higher than that remaining in the presence of dispersant. Indeed, in this case the suspension has less affinity for the support than when it contains dispersant due to the light modification of the surface tension.

### 3.4 Estimation of the adhesion/attrition mechanism

The adhesion represents the tendency of the powder particles to stick to each other or to the inert particles. The performance of the drying process in a fluidized bed of inert particles can then be estimated by the propensity of dried coating to peel off from inert particles and by avoiding the formation of

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![Fig. 12](image1.png)

**Fig. 12** Influence of the drying temperature on the drying kinetics. 
($Q_{\text{air}}=50$ L/h, 1% PNa$_{5000}$, untreated plates)

![Fig. 13](image2.png)

**Fig. 13** Influence of the plate hydrophobicity on the drying kinetics. 
($T=65^\circ\text{C}$, $Q_{\text{air}}=75$ L/h, 1% PNa$_{5000}$)
agglomerates. This phenomenon depends on the attrition of the glass spheres related to the fluidization movement, but also to the adhesion forces between the dried coating and the inert support.

The shearing tests show the same tendency for the specimens made by only one or two coated plates (Fig. 15). The shear modulus is maximum for highest concentrations of dispersant and for lowest humidity. Indeed, dispersant addition tends to decrease the size of the calcium carbonate particles by deflocculation of the agglomerates. This makes it possible to increase the surface energy and thus the energy of adhesion. The influence of the dispersant content can also be related to the surface-active character of sodium polyacrylate. It improves the wettability of glass and thus the adhesion of the suspension on the support.

In addition, when the humidity increases, the powder flow, i.e., the displacement of dried film, is increasingly difficult because the increase in the water content of the powder involves the creation of interparticle liquid bridges (Teunou, 2003).

To decrease the shear stress and to limit the agglomeration and the sticking of glass particles in the process of drying in a fluidized bed of inert particles, it is necessary to increase the relative humidity. However, the process being continuous, a high humidity always remains in the fluidized layer.

Moreover, to improve the peeling of the dried powder and its recovery, it is necessary to optimize the quantity of dispersant: sufficiently high to make the suspension sprayable, but sufficiently low to reduce the affinity of the film for the coated surface.

4. Conclusions

The present work involved the preparation and spray drying of highly concentrated suspensions of calcium carbonate. The effectiveness of polyacrylate salts on the reduction of the viscosity was confirmed for the suspensions of calcium carbonate.

Drying of these concentrated suspensions by two spray drying methods showed that it is possible to form particles of different shape and size depending on the method used. It was also shown that the drying in a fluidized bed of glass spheres can be applied to concentrated slurries with a low viscosity. Conventional spray drying of such suspensions leads to
granules, whereas drying on fluidized inert particles enables a fine powder close to the elementary particles to be obtained.

From the study of the drying kinetics, we were able to highlight the influential parameters on the kinetics of drying. It appeared that the use of a hydrophobic support could be beneficial provided that the suspension is finely sprayed at the surface of inert particles (i.e. by using a pneumatic nozzle).

In addition, surface tension of the suspension and ambient humidity seem to be crucial parameters to optimize the powder recovery after drying the coated layer.

These results extrapolated on the level of the coating of glass spheres make it possible to better understand and optimize the process of drying in a fluidized bed of inert particles.

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Nomenclature

| Symbol | Description | Unit |
|--------|-------------|------|
| B      | Blaine specific surface | cm²/g |
| d      | displacement | m |
| e      | porosity |   |
| E      | thickness | m |
| F      | load | N |
| G      | shear modulus | Pa |
| K      | Blaine apparatus constant |   |
| l      | specimen length | m |
| L      | specimen width | m |
| LOD    | Loss On Drying |   |
| Q      | flow rate | m³/h |
| RH     | relative humidity | % |
| S      | specimen surface | m² |
| t      | time | s |
| T      | temperature | °C |
| U      | air velocity | m·s⁻¹ |

Greek letters

| Symbol | Description | Unit |
|--------|-------------|------|
| ε      | strain | [-] or [%] |
| γ      | shear rate | s⁻¹ |
| η      | viscosity | Pa·s |
| ρ      | density | cm³/g |
| σ      | stress | Pa |

Subscripts

| Symbol | Description |
|--------|-------------|
| c      | shear |
| f      | fluidization |
| i      | inlet |

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