

# CHARACTERISATION AND REDUCTION OF 'WALL STICKING' IN SPRAY-DRYING CLAY SUSPENSIONS

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## ABSTRACT

*So-called 'wall sticking' consists of the accumulation of suspension drops, general partly dried in flight, on the inner walls of the spray dryer. On an industrial level, this effect usually alters the size distribution of the granules made, and causes accidents and production shutdowns due to the collapse of slabs of agglomerate powder. Wall sticking often appears due to erroneous handling of the equipment, either because of attempting to dry more slurry than originally foreseen in the spray-dryer design, or because of a bad mixture of the jets of hot air and drops of slurry in the upper part of the tower. The effect of these parameters is discussed on the basis of an industrial case study where the spray dryer of a tile plant was used above its production capacity. The generated amount of wall sticking was measured, and the affected spray dryer surface was estimated. Simple corrective measures were implemented, which enabled reducing this undesirable effect by up to 92%. On the other hand, the factor 'raw material' has a special relevance, since the surface permeability of the drops dried in flight, and therefore, their consistency when impinging against the spray-dryer walls, can depend on the raw materials used, and the additives and soluble salts present. At laboratory level, drops of various raw materials were sprayed, with the same size and density, in the same drying air, projecting them onto metallic substrates at ambient temperature, or preheated to 120 °C. In each case, residual moisture, adhered material/impinging material mass ratio ('deposition efficiency'), and the adhesion of the clayey layers on exposure to shear or tensile stresses by centrifugation (1100 g), were measured. The influence of the submicron material fraction on the mechanical behaviour was noted, as well as the effect of substrate temperature and the type of applied stress, thus contributing to explain some of the differences in behaviour between the layers formed on the spray dryer vertical wall and on the spray dryer conical wall.*

# 1. INTRODUCTION

Many companies dehydrate aqueous suspensions (foods, different stoneware suspensions for tiles, chemical products, medicines) by means of the so-called ‘spray-drying’, technique which consists of pulverising the suspension in small drops and drying these in flight by a jet of hot air, in a cylindrical tower with a conical end (figure 1a). The solvent used to produce the drops evaporates in flight, progressively, within the tower, leaving granules with low residual moisture.

Theoretically, all the drops should manage to dry suitably, fall, possibly bouncing against the lower (conical) wall of the tower, and be recovered immediately to be sent to the rest of the manufacturing process (storage and pressing, or conditioning and despatch). In fact, tile manufacturers try to obtain an optimum size distribution for these granules (almost spherical), in order to press more compact and resistant green tiles<sup>[1]</sup>. However, in practice, it is frequently observed that the drops dried in flight, instead of bouncing and being immediately recovered as dry spherical granules, are retained on the wall, for times that can vary between some seconds and several days. The retained material subsequently falls, in the form of dry or semi-fired aggregates, depending on the temperature of the impact area (figures 1c, 4b), comprising various granules with sizes that can vary from some hundreds of micrometers to several tens of centimetres, and which, definitively, does not correspond to the desired size distribution for optimum pressing<sup>[2]</sup>. When thick slabs form, the effect of gravity or vibrations causes sudden falls (collapses), thus interrupting the production process. In some cases, labour accidents can occur (burns, and even explosions, in the case of handling organic substances, as is the case in the dairy industry).

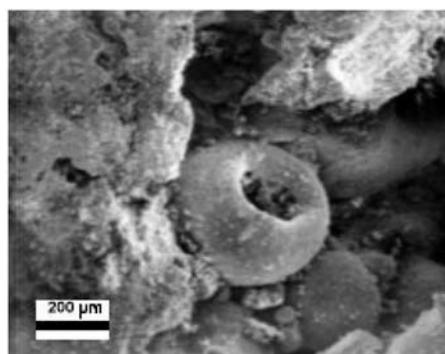
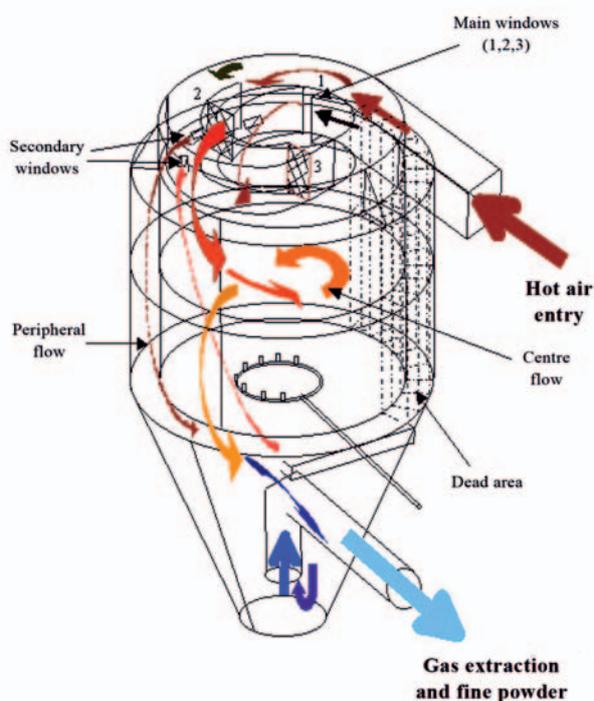


Figure 1. (a) description of a fountain-type spray dryer type (b) internal view of the hat and of a secondary window (peripheral flow) (c) example of wall sticking with the case of a granule completely dried in flight sticking, however, to wet deposits.

## 2. IMPORTANCE OF THE RAW MATERIAL USED IN IN-FLIGHT DRYING AND ADHESION

The main cause of this effect is the incomplete evaporation of the solvent in the drops. At the level of the raw material used, two elements of drop behaviour, linked to their composition, can increase wall sticking: (a) low permeability of the drop surface formed in flight, causing it to keep a greater moisture when impinging on a wall, and (b) after the impact, a greater capacity to adhere, by means of greater substrate-drop contact<sup>[3,4]</sup> and the formation of capillary bridges<sup>[5,6]</sup>.

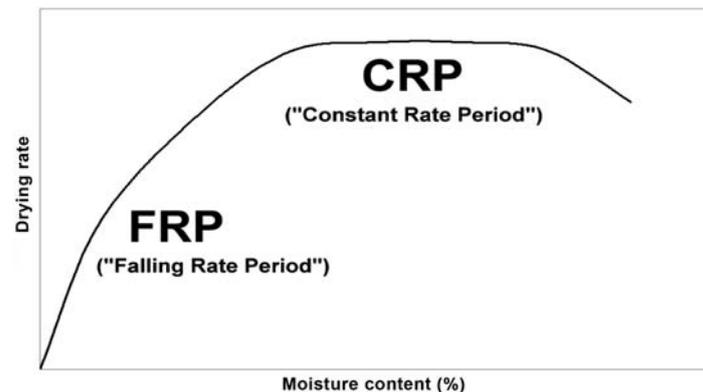


Figure 2. Classic description of the drying phases in spray drying

When drops dry in flight, they lose water in their interface with the drying air, i.e., at their surface. In a classic approach<sup>[7]</sup> (figure 2), the existence is assumed of a preliminary drying period (CRP) with constant-rate evaporation, in which the drop behaves as if it consisted of pure water, whose evaporation is not altered by the presence of solid particles from the suspension. This phase is followed by a period (FRP) in which the solid particles concentrate in the surface and hinder the migration and evaporation of the water, now totally depending on the permeability of the formed shell. In this sense, under equal spray-drying conditions, two slurries can give rise to different residual moistures: the slurry that generates a drop shell which is only slightly permeable will be more prone to wall sticking. This permeability is linked to the particle size distribution of the clay mixture, after the Carman Kozeny formula:

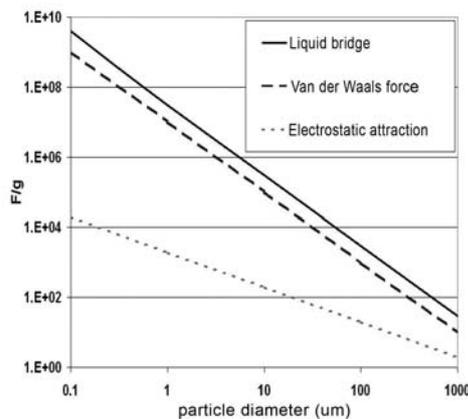
$$K = \frac{\varepsilon^3 \cdot d^2}{A \cdot (1 - \varepsilon)^2}$$

where  $\varepsilon$  is the porosity of the medium,  $d$  the diameter of solid particles (assumed spherical.  $A=60-180$ ). Note that a finer and more compact texture gives rise to a lower permeability, which is why the morphology of the particles (often non-spherical) of a mixture for tiles, their piling, as well as the presence of molecules adsorbed onto their surface, will be elements to be taken into account. For example, a totally deflocculated suspension produces a more closed and less permeable texture than its equivalent partly deflocculated one<sup>[8-10]</sup>. On the other hand, the presence of soluble salts, such as sulphates or chlorides, usually gives the suspension a gelatinous texture with a significant amount of water bound to clay micelas, which is why free water migration depends on the pressure to which it is subjected<sup>[10,11]</sup>. This phenomenon takes place especially when much water is left to evaporate and the inter-particle distance remains

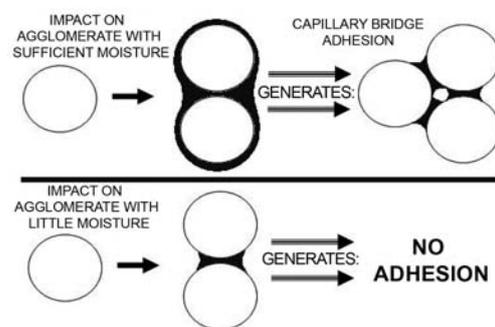
high, which obliges adjusting the concept of hindrance-free drying in the initial drying phase ('CRP').

Depending on the case involved, the objects obtained in impingement can be completely squashed drops, or semi-dry domes<sup>[3]</sup>, or granules adhered without deformation (figure 1c, 4b). In effect, viscosity (and hence, the difficulty of deforming under impact) of a drop grows exponentially when lowering its residual moisture. That is to say, its tendency to adhere instead of bouncing increases with its surface moisture, because the deformation of the drop is favoured and a greater contact surface develops<sup>[6]</sup> with the substrate or the already formed clay layer. This surface moisture is not only tied to the permeability of the drop shell, but also to the value of the moisture gradient that occurs in this shell. In this regard, a slightly deflocculated suspension rich in sulphates presents a low moisture gradient at its surface<sup>[10]</sup>, whereas the same suspension, poor in sulphates and totally deflocculated (minimum viscosity in the deflocculation curve) displays greater moisture gradients. For this reason, although both suspensions reach the wall with the same residual moisture, the drops of the second suspension have less moisture in the surface, making them less prone to deformation and adhesion.

Although it is possible for objects to adhere without moisture, by means of Van der Waals or coulombic interactions<sup>[4,12]</sup>, the specific adhesion force ( $F_s = F / (m \cdot g)$ ) where  $F$  is the adhesion force,  $m$  drop mass and  $g$  the earth's gravity) generated by liquid bridges, predominates (figure 3a) in these interactions, when there is a sufficient amount of water<sup>[6,13]</sup> (pendular state). This is the reason for the relevance of residual moisture. Once the drop has made contact, the water present (from the surface of the impinging drop or in the layer of wall sticking itself) can form bridges between the impinging particle and the surface (metallic or already soiled), as Schaafsma et al. state<sup>[5]</sup> (figure 3b). Note that a dry granule that enters into contact with a sufficiently wet surface can remain adhered by this mechanism (see figure 1c). For this reason, it is advisable to keep the agglomerates formed on the walls from being made up of 100% badly dried drops. However, below a certain level of moisture, the liquid bridge mechanism can stop working, and the impinging particle is more prone to detach, as it depends more on alternative adhesion mechanisms<sup>[6]</sup>.



(a)



(b)

Figure 3. (a) comparison of the different adhesive forces (sphere-sphere configuration)<sup>[6]</sup>, considering a density of 1800 kg/m<sup>3</sup> (b) liquid bridge formation mechanisms according to Schaafsma et al.<sup>[5]</sup>

As far as the specific adhesion force is concerned, depending on the interaction mechanism involved<sup>[14]</sup>, the effect of particle size can increase or reduce this, which is why the literature (see the comparison presented by Lam and Newton<sup>[15]</sup>) displays contradictory tendencies on this point. However, the contribution of the liquid bridge mechanism between smaller particles gives rise to a greater specific adhesion force<sup>[13]</sup>, which is why the finest raw materials could be more bonding. On the other hand, a certain particle size distribution can increase the compactness of the construction formed<sup>[11]</sup>, giving rise to a denser and more resistant deposit in the face of the possible stresses that may be exerted on the layers formed.

These adhesion-opposing stresses may be generated by:

- (a) **Viscous friction of the drying air** (in laminar or turbulent flow) on the wall sticking surface<sup>[16]</sup>: this effect is probably most noteworthy by the detachment of small aggregates of granules, especially the driest.
- (b) **Gravity**, which mainly contributes to the detachment of thick slabs and blocks, when shear stress predominates over the cohesive and adhesive stress of the capillary bridges and the solid friction between clay layers. However, note that on the conical wall of the spray dryer, the weight of the forming layers involves, in addition to a shear component, a perpendicular component that contributes to compacting the layers, increasing their intergranular cohesion, and delaying their detachment until the shear component is sufficiently high to overcome the adhesion (figure 4a). The value<sup>[6,12,14,15,17]</sup> and the time<sup>[18]</sup> of application of this external preliminary compaction force ('press-on force') can drastically increase the value of the adhesion force of the deposited layer. This would explain why a greater wall sticking thickness is usually found in the conical area of the spray dryer.
- (c) **External mechanical stresses**: they include the vibrations that the machinery undergoes, intentionally or not, as well as the cleaning actions *in situ*, by means of pressurised water or with metal tools (dry cleaning option). These latter tools are used periodically in the companies careful of the maintenance of their spray dryer, dry cleaning being the most efficient option, although also the most difficult in its manual version, and, in its automated version<sup>[19]</sup>, most cumbersome and least used.

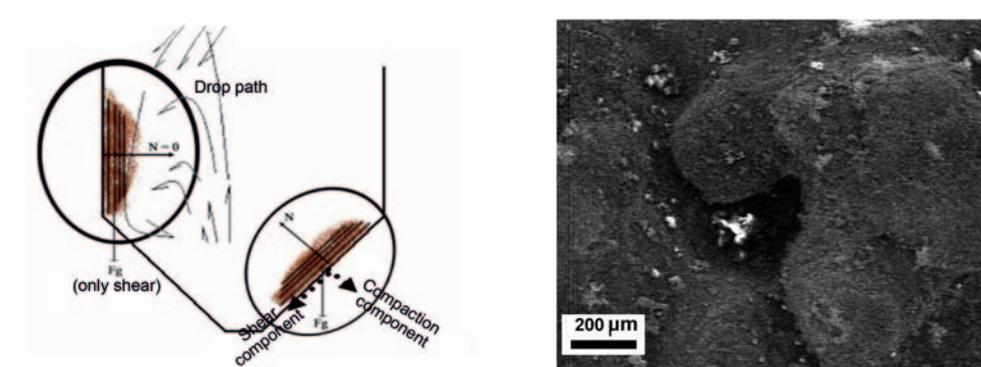


Figure 4. (a) influence of the position of wall sticking in the tower, on the type of applied stress  
(b) detail of capillary bridges formed between granules (micrograph, SEM Jeol T300)

The foregoing indicates the importance of the position of the wall sticking in the spray-dryer tower. In this respect, also to be taken into account is the wall temperature: the vertical part is subject to temperatures above water boiling temperature (which evolve from the production cycle start until the steady work regime). Logically, vertical wall sticking includes not very thick layers with a porous texture (rapid expulsion of water by boiling)<sup>[3,20-22]</sup> and even semi-fired parts owing to the high wall temperature. The conical area is subject to lower temperatures, which is why the effect of boiling on impact is less relevant. On the other hand, the drops that hit the conical wall have had more time than in the previous case for the free water to evaporate in flight: the semi-wet texture (pendular state) then predominates, with the presence of capillary bridges<sup>[5,13]</sup> between the objects (Figure 4b).

It may be noted that, despite the frequent appearance of so-called ‘wall sticking’, this is barely documented for the tile industry<sup>[2,12,23-24]</sup>, and the literature usually attaches greater emphasis to the adhesive effect of the dry organic components (lactose, starch, others<sup>[4,11,15,25]</sup>), where Van der Waals or coulombic interactions predominate), than in the still wet atomised suspensions (where the interactions generated by liquid bridges are more relevant<sup>[6]</sup>).

### 3. IMPORTANCE OF SPRAY-DRYER HANDLING

Other key factors<sup>[26]</sup>, like the type of machinery, or the way of using it (‘method’ and ‘labour’ factors) have received little attention. The ceramic tile industry only uses inorganic suspensions, which form deposits whose moisture approaches the pendular state, and needs to resolve the problems of wall sticking without significantly altering the manufacturing process. In this sense, a study of the factors linked to the use of the machinery, such as drop injection and drying air distribution, is the most useful to start with. The comparative effect of clays and their own adhesion become important once this first point has been solved.

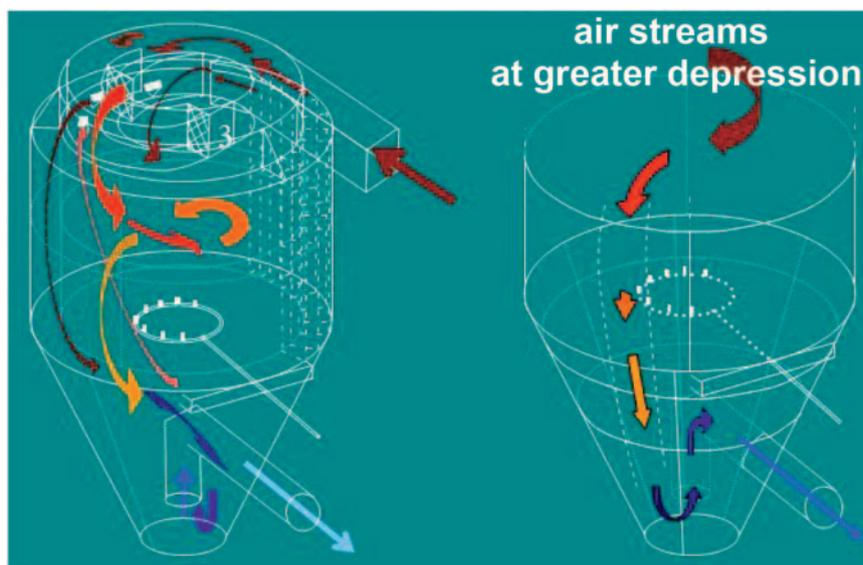


Figure 5. Effect on air and on drop circulation of a strong depression value at the extractor level: the air is renewed more quickly and maintains low moisture, but the drops remain less time in the drying area.

It is logical to consider that a spray dryer used above its capacity (sending an excessive flow of suspension or a suspension too rich in water) will be more prone to wall sticking. Certain companies eager to increase their production prefer, for tactical and economic reasons, to take this risk instead of investing in an additional spray dryer. Of course, the possibility exists of compensating the shortage of hot air with the strong flow of suspension, by increasing the power of the pressurisation fan, the burner and/or the extraction fan. However, this option is very prone to wall sticking, whenever the drying tower has little volume for the (increased) exchange of mass and heat. Instead of having a greater exchange space, the option remains of trying to increase the exchange time, controlling the air circulation velocities in the tower (extraction and pressurisation flow rates, respectively), but the air present can become excessively damp and lose its drying capacity (figure 5)

Apart from the considerations of the drying capacity designed by the constructor of the facility, the approach of mixing the volume of liquid with the volume of hot air that reaches the spray dryer requires particular care.

A spray dryer consists of a generator of hot air tangentially directed towards the high part, called the 'hat', of the spray dryer. A series of main windows (adjustable openings) and secondary windows (fixed openings: figure 1a, 1b) distributes this air towards the cylindrical part of the tower. This air is distributed and cooled as it circulates within the hat, which is why this air, which descends in a spiral, cannot reach all the drops impelled by the injector crown with the same temperature and the same flow rate. Consequently, it is recommendable to adjust the amount and size of the drops that are sprayed into the contacting air: if very large and/or numerous drops are sent into a sector of the spray dryer where the drying air arrives with a low flow rate or temperature, drying will be imperfect and give rise to wall sticking.

In this respect, the arrangement of the injectors in the crown of the fountain-type spray dryer must take into account, and even avoid, this encounter of the drops with air that is very inefficient: although an optimum area exists in which to impel the wet drops, there is also what may be called a 'prohibited injection area' (sector of the crown where the sprayed drops will make contact with very little drying air), and of a 'reserve injection area' (sector where these drops will have an incipient contact with a more important air jet: see figure 6).

Note that the spiral path of the air also contributes a rotational component to the trajectory of the drops, which is why there should be a staggering between the angular position of the drops when they leave the injectors and their angular position when they reach the upper part of the tower: the drops of the so-called 'prohibited injection area' do not travel vertically and, in fact, a deposit will be observed in another sector of the wall, which we could call the 'dead area' (very little hot air circulation), located between the last window (less hot air) and the first window of the spray dryer (more hot air). The drops expelled from the crown into the so-called 'reserve injection area' sector (practically in the vertical of the 'dead area') will travel, by means of the rotational component of their trajectory, towards the exit of the first window, which provides very hot, efficient air. However, the first part of the displacement of these drops will occur via the dead area space, which is poor in drying air.

In contrast, it is recommended to send the largest and most numerous drops to the air that leaves the first windows, as its drying power is highest. In short, the so-called 'crown plan' (where to place injectors, and with which opening diameter?) is a

crucial parameter, not only for defining the granule amount and size distribution, but also to optimise drop drying.

On the other hand, it is recommendable that the path of the largest drops (more difficult to dry owing to their low surface/volume ratio) should be associated with a greater residence time in the drying air and, therefore, that these drops be driven to greater height in the tower. In this sense, it is possible to increase the injection pressure and the height of the spirals ('ringlets') within the injectors <sup>[27]</sup>, which enable higher flight (with greater exposure to the drying air) of the drops.

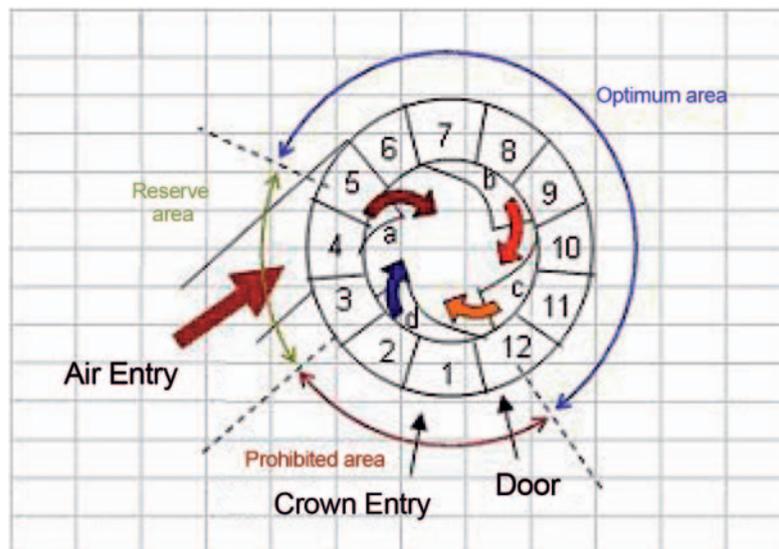


Figure 6. Arrangement of the four main windows and zoning proposed by sections of the internal wall, in the spray dryer used in the present study. The so-called 'prohibited' and 'reserve areas' for the installation of the injectors in the crown are indicated.

The solution of the problem of wall sticking in tile plants involves two approaches: first, at plant level, by measuring and reducing wall sticking, adjusting the distribution of air and drops in the spray-drying tower; and secondly, at laboratory level, for which methods are proposed to compare the adhesion of suspensions of different raw materials deposited in equal conditions.

#### 4. TREATMENT OF THE PROBLEM ON AN INDUSTRIAL SCALE

The first approach of the present work was undertaken at an industrial spray dryer, theoretically designed to dry 5100 kg water/h, and modified by a Venezuelan tile company, in order to increase its production of granules from approx. 12000-14000 to 18000-19000 kg/h. Although the power of the hot air generator had been increased (9.000.000 kcal/h instead of 5.000.000) as well as that of the extraction fan (188 HP instead of 75), the volume of the spray drying tower remained the same, and wall sticking became catastrophic.

First, a methodology was elaborated to evaluate how important the phenomenon to be studied was:

- **Mass method:** the adhered material that falls between two cleaning periods, spontaneously, is collected in a box outside the spray dryer, and its weight is

evaluated. The advantage of this method is the possibility of easily measuring the produced sticking, without needing to enter the spray dryer. However, it does not include the smaller-sized aggregates, and provides an incomplete view of the formation of layers in the spray dryer.

- **Mapping method:** observation *in situ*, to develop a thickness map, is a useful complement of the mass measurement method mentioned above. This mapping method enables locating the sectors of the tower that are more prone to the generation of sticking, and differentiating the produced types of sticking. There are two measurement modalities:
  - **Measurement of thickness:** this approach consists of entering the spray dryer (figure 7) and measuring *in situ* the thickness of the layers of sticking formed. This method is consistent with the procedure used in some plants for periodic cleaning of the spray dryer, as an operator is usually sent in with a metal tool (*chicura*) for the dry removal of the thickest layers. The operator can take advantage of this operation to measure the thickness by means of a graduated bar. This method is particularly useful for characterising the thickest layers, but it is difficult to measure the thicknesses in all the internal surface of the spray dryer, in addition to the relative danger of detachment that exists with this procedure. Finally, in the case of the plants that only use pressurised water jets for cleaning, this procedure does not apply, although it has been observed that cleaning just with a water jet is not wholly efficient.
  - **Visual estimation of thickness:** a visual appreciation of layer thickness on the basis of a comparison with the results of the previous approach has been proposed. This approach enables quite a rapid and much less hazardous study than direct measurement, although it is less accurate (5-10 mm) and depends on the skill of the operator.



Figure 7.

Direct measurement (a,b) or visual estimation (c) of thickness in thick and thin layers of wall sticking.

The corresponding map is made by dividing the internal periphery of the tower into sections (abscissa of the graph), and by dividing the height of the tower in levels (ordinate of the graph: see figure 11). On the other hand, observation *in situ* enabled distinguishing several types of sticking formations (figure 11):

- (A) **Vertical wall sticking:** consists of drops that are still very wet impinging against the vertical walls, especially in the 'dead area'. They display a compact

texture in their primary layer, but a more porous and crumbly appearance in the following layers. The characteristic colour is reddish, due to the high temperatures present.

- (B) **Cone wall sticking or 'ball sticking'**: This forms when the semi-dry drops hit the walls of the conical base, and accumulate according to the mechanism described by Schaafsma et al.<sup>[5]</sup>.
- (C) **Extractor Tube Sticking**: this forms when the air streams push and project the wet drops of slurry onto a side of the extraction tube. It is observed directly inside the conical chamber during a shutdown of the spray dryer; and when there is production, by the outlet in the form of dry lumps with a laminar morphology (with layers 3-6 mm thick), which fall suddenly, in some cases blocking the powder exit.
- (D) **Crown sticking**: this forms when the liquid drops are projected towards the crown by the air stream, or when the injectors leave liquid slurry on its surface. Compact and dense agglomerates are produced, especially when the spray-drying cycle begins, because spray drying is imperfect at the beginning of this cycle. In effect, the slurry that leaves when the injection pumps start does not become individual drops immediately, which is why the liquid pours out in a continuous jet, and falls onto the crown by gravity. Coarse dry agglomerates can form, very similar to those of 'extractor tube sticking', located on the elbow of the extractor cone, but without the laminar structure.
- (E) **Crown tube sticking**: Just as extractor tube sticking, it is very difficult to remove, but it differs from this by its location, as it tends to generate a line of material deposited in the conical part of the spray dryer. It has a very wet and plastic appearance, and is hardly 2-4 mm thick when observed from the outlet.

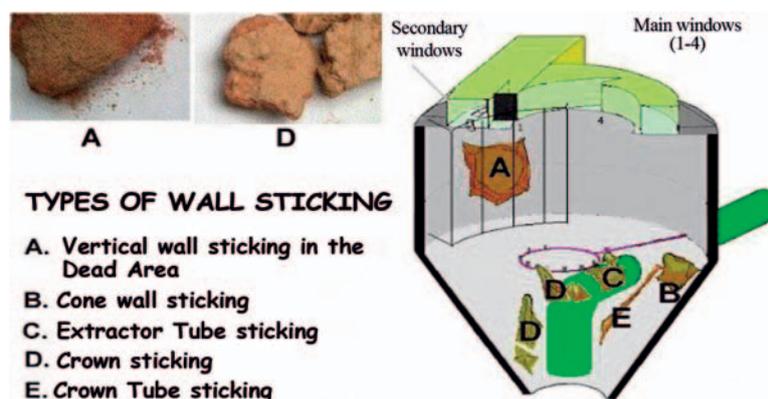


Figure 8. Location and appearance of the different types of sticking formed.

The study to reduce wall sticking was undertaken with care to maintain a production of 18,000 kg granules/h, with residual moisture and particle size conforming to the requirements of the plant. In each adjustment, the parameters indicated below were modified, and the mass and mapping measurements were made:

- powers of the extraction and pressurisation fans, measuring the depression level (mm H<sub>2</sub>O) at the level of the extractor tube

- opening of the main windows (the first three are adjustable by levers located in the ceiling, whereas the last is open)
- injection pressure of the slurry
- arrangement of injectors in the crown ('crown plan')

## 5. COMPARISON OF RAW MATERIALS ADHESION IN THE LABORATORY

The second approach, linked to the tendency of a raw material to adhere more or less to the walls, required the reproducibility conditions of a laboratory. In this sense, different common raw materials were used (table 1), put into suspension in similar density conditions (70% solids by weight), viscosity (Brookfield RVF), deflocculation (i.e. possible minimum viscosity, obtained with sodium tripolyphosphate). Their particle sizes (Micromeritics Sedigraph) are represented in figure 9. The other characteristics mentioned in the present work were measured and are presented in the reference<sup>[10,28]</sup>.

RAW MATERIAL	TYPE	ORIGIN	DEFLOCCULATED VISCOSITY (CPO)	SULPHATE CONCENTRATION (ppm)
K	Kaolin	U.S.A.	100	230
8	Kaolin	Bolivar	525	633
A	Illite	Lara	50	426
F	Feldspar	Cojedes	-	31
D	70% A + 30% F	-	50	308

Table 1. Characteristics of the raw materials used.

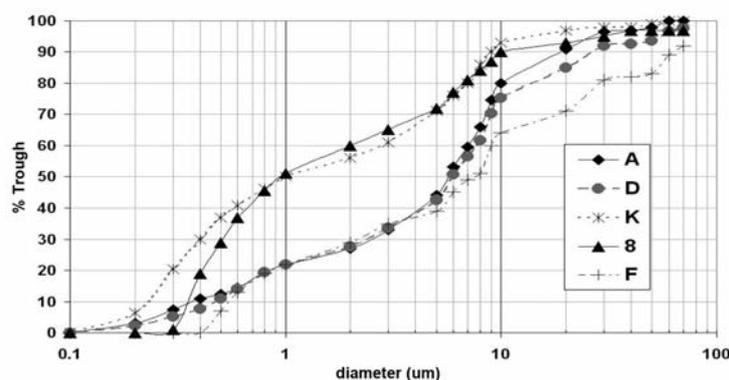


Figure 9. Particle sizes of the mixtures used.

The suspensions were projected ('sprayed') by successive volumes of 10 ml, with an assembly (see figure 10a) involving a SATA Minijet 90 airbrush gun, whose air and liquid flow rates were controlled in such a way as to hold the distribution of the drops size across the different suspensions, following the Kim-Marshall formula<sup>[24]</sup> and taking into account the values of the hydrodynamic characteristic (relatively near

to each other with the exception of mixture 8, which was very rich in sulphates) of the suspensions:

$$D_{MMD} = \left[ \frac{249 \sigma^{0.41} \mu^{0.32}}{(V_a^2 * \rho_a)^{0.57} * A^{0.36} * \rho_l^{0.16}} \right] + 1260 \left[ \left( \frac{\mu_l}{\rho_l \sigma} \right)^{0.17} * \left( \frac{1}{V_a^{0.54}} \right) * \left( \frac{M_a}{M_l} \right)^m \right] = 50 \mu\text{m}$$

Where:

- $D_{MMD}$ : mean mass diameter of the distribution ( $\mu\text{m}$ ),
- $\rho_a$ : density of the air ( $\text{lb}/\text{ft}^3$ ),
- A: Surface of the air ring in the nozzle ( $\text{in}^2$ ),
- $\mu$ : suspension viscosity,  $\sigma$  suspension surface tension;
- $M_l$ : liquid mass flow rate ( $\text{lb}/\text{min}$ ),
- $M_a$ : air mass flow rate ( $\text{lb}/\text{min}$ ),
- $m = -1$  if  $(M_a/M_l) < 3$ , otherwise,
- $m = -0.5$ .

The pulverisation air was heated to  $50^\circ\text{C}$  by electric resistances, a temperature regulator, and a heat exchanger. The aerosol formed and dried in flight over a controlled distance, fell on metallic steel substrates AISI 304, previously cleaned with alcohol. All the tests were conducted on horizontal substrates (5 for each case) at ambient temperature located at 50 cm distance from the gun. In addition, deposits of mixture D were prepared on substrates preheated at  $120^\circ\text{C}$  (test 'DD').

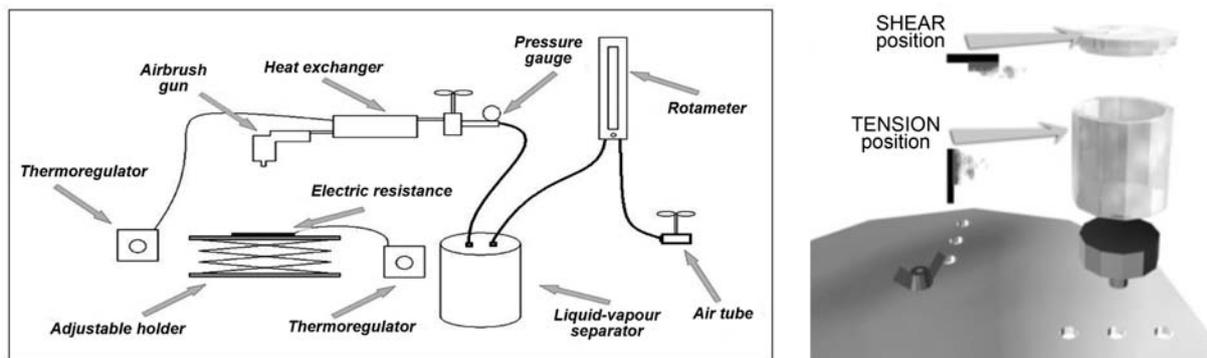


Figure 10. (a) principle of the assembly used to deposit the clay layers and (b) sample holder for applying shear or tensile stresses to the deposited layers.

The residual moistures of the layers deposited on the substrates were determined by means of an analytical balance (Mettler), and the deposition efficiency was determined dividing the solid mass fixed on the surface of the substrate by the mass theoretically projected towards this substrate. In this sense, the conical shape of the aerosol was taken into account, as well as the Gaussian distribution of the sprayed mass to evaluate the amount projected onto the substrate, according to the method described in<sup>[3]</sup>.

The measurement of clay deposit adhesion was made with a Hettith EBA35 centrifugal machine, modified to generate accelerations up to 1100 g. The force of centrifugation on the formed deposit is given from  $RCF = (r_r \cdot \omega^2) / g$ , where  $r_r$  is the rotation radius and  $\omega$  the angle velocity. This centrifugation technique is mentioned in the literature by a small number of authors <sup>[12,15-18,29]</sup>, and its relation to the industrial problem of wall sticking (except the work by Rennie et al.<sup>[12]</sup>) is hardly evoked. In addition, all the authors mentioned apply a preliminary compaction ('press-on') of the powder, whose moisture stays very low voluntarily, in air with a relative humidity below 50%: this condition forces the study to focus on Van der Waals adhesive mechanisms, instead of capillary bridges (more relevant in industrial spray dryers). In the present case, the layers formed reached moistures between 7 and 16%, which is why the pendular state was at least assured. 'Press-on' was not applied, and the effect of the weight of the successive layers and of the propulsion air pressure of the airbrush gun was, theoretically, of little significance.

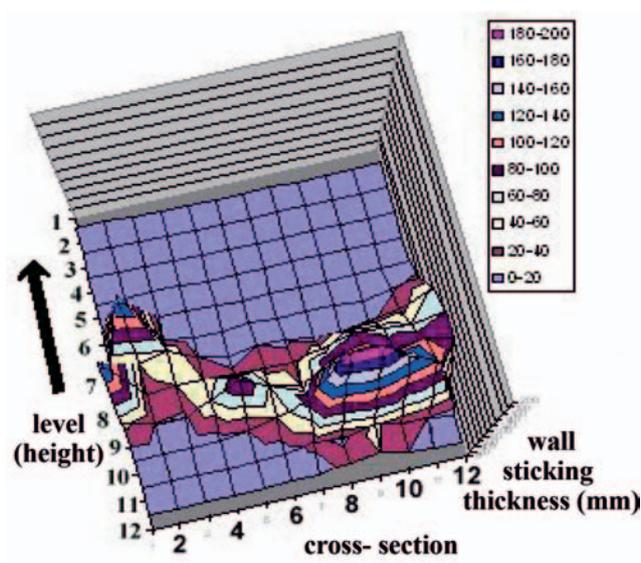
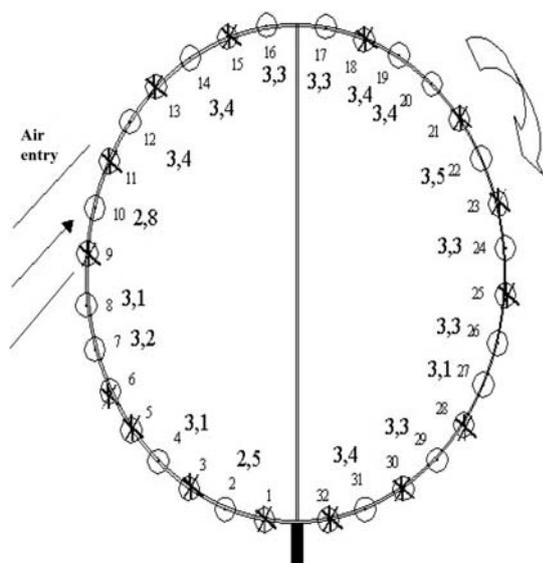
On the other hand, all the adhesion measurement tests by centrifugation mentioned in literature apply the centrifugal acceleration vector perpendicularly to the surface of the covered substrate, applying, according to the application direction, the 'press-on' effect or, with an upside sample holder, the tensile stress. However, in reality the stresses applied to wall sticking in industrial spray dryers correspond essentially to shear stresses. For this reason, the sample holders employed in the present work were designed (figure 10b) to simulate both tensile stresses and shear stresses (vector acceleration coplanar to the surface of the substrate). In both cases, the acceleration was applied for 7 minutes (after which time no more loss of material was observed), and the percentage of dry mass lost was measured as an indication of the poor adhesion of the deposits. The centrifugal machine, by its reduced rotation rate (3300 rpm), did not allow measuring the acceleration necessary to remove all the deposited material. However, the tests performed at the maximum capacity of the machine enabled comparing the behaviour of the different studied clay mixtures.

## 6. REDUCTION OF WALL STICKING IN AN INDUSTRIAL SPRAY DRYER

**Initial configuration:** summarising, it can be observed in figure 11 that the essential part of the injected hot air is sent via the 4th main window, which causes a significant amount of air to leave by the secondary windows that exist in the hat, which explains the very moderate amount of sticking on the vertical walls (levels 1-4, although a significant amount is observed on level 5). However, the air distribution in the centre of the tower (air issued through the main windows) is not very homogeneous or efficient, which is why a very important production of wall sticking is observed (over 300 kg/h), especially at the level of the conical walls. The 'crown plan', i.e. the arrangement of the injectors in the crown (and their respective openings), does not take into account that only one part of the air will be really effective in drying the projected drops. On the other hand, a particularly thick deposit is observed in sections 8-10 of level 10 (conical part). It was noticed that this deposit happened especially at a place in the spray dryer where the extractor tube was eccentric, the deposit being observed in the area where there was more room between the tube and the wall: it is assumed that the slower air facilitates formation and accumulation of material. For this reason, it was attempted to centre the extractor tube again (figure 12). On the other hand, it was considered that a greater slurry injection pressure could provide the drops with more opportunity to dry in flight, by having a greater projection height in the tower and, thus, produce less conical wall sticking (ball sticking). Finally, it was proposed to increase the level of

extraction in the extractor (greater depression value) to reduce the degree of moisture of the air in the tower and provide the drying air with greater efficiency.

OPENING OF THE MAIN WINDOWS	% PRESSURISATION	% EXTRACTOR	INJECTION SUSPENSION	MEASUREMENTS
1st: 0%, 2nd: 10%, 3rd: 10%	100 %	90 %	20 bar	Depression: -5 mm H <sub>2</sub> O Sticking emission: 323 kg/h



Sum of diameters: 54.8 mm; Sum of areas: 139.5 mm<sup>2</sup>

Note: the used spray dryer has one fourth (last) window, always completely opened

Figure 11. Service conditions (configuration selected by the company, after upgrading the spray dryer); surface map of wall sticking and mass production of wall sticking.

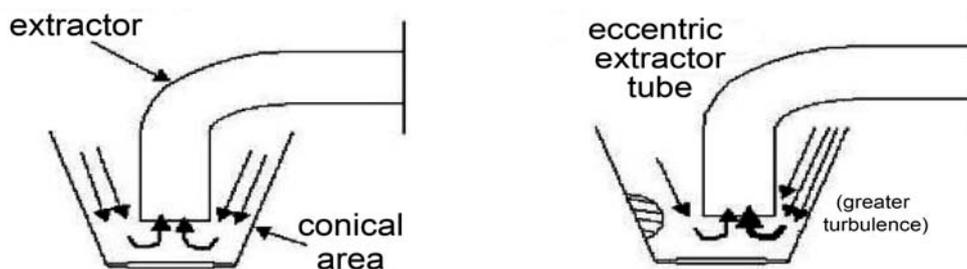


Figure 12. Observed effect of a centred or non-centred position of the extractor tube on wall sticking.

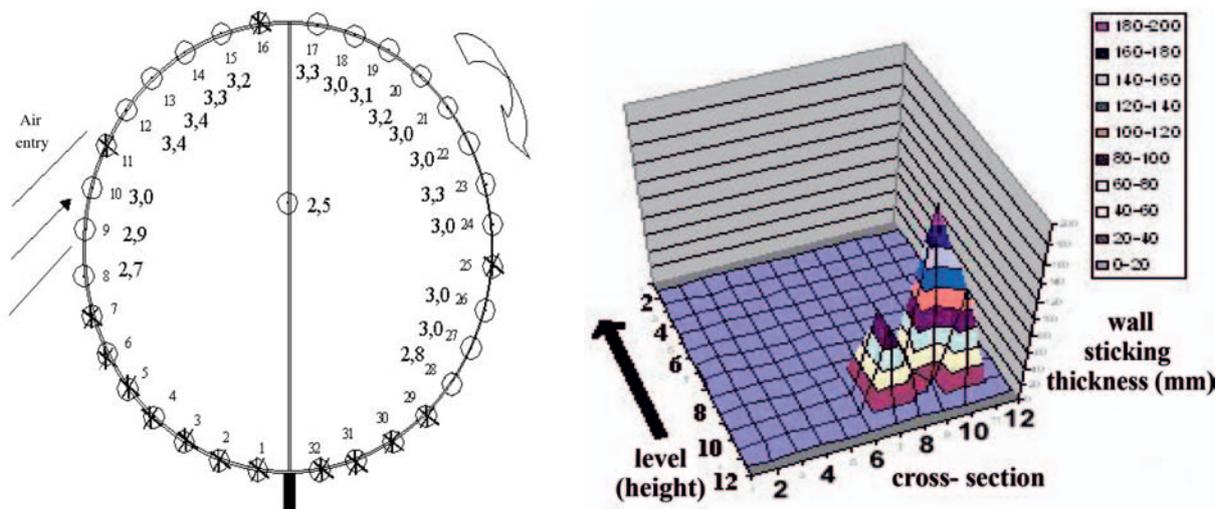
**Adjustments:** while reducing the air flow rate in the spray dryer, this air was simultaneously used more efficiently, opening the first main windows more in order to distribute the hot air better on all the angle sections of the central volume of the tower. In parallel, the crown plan was arranged in such a way that there were no injectors in the so-called 'prohibited areas' and 'reserve area'. The injection pressure was increased by 1-2 bars, and the depression in the extractor from 3-5 mm H<sub>2</sub>O. The improvement

was immediately noticeable, as the recorded sticking mass produced was reduced to a tenth. However, significant deposition continued to be observed in levels 5-7, section 1. As section 1 corresponded to the area where enters the air by main window no. 1, it was concluded that there was still a deficiency of drying air near this window. For this reason, this window was opened further, producing an immediate improvement.

In regard to the greater depression value used, the following effect was observed: the form of the sticking that appeared in the conical part of the spray dryer displayed very steep spiral lines (vertical), like 'twisted straws'. This suggested that the drops travelled less time in the tower, and this short residence time limited the capacity of the air to dry the suspension drop (figure 5). For this reason, an intermediate depression value prevailed, which also enabled reducing (until eliminating) the formation of sticking at levels 6-7, without wholly eliminating them at level 10. On the other hand, in the spray-dryer start-up routine, water was injected at the start instead of slurry, to limit crown sticking.

Overall, the distribution of the windows and the crown plan seem to be the most determining elements for the success obtained.

PARAMETERS	BEFORE	AFTER
Opening of the main windows	1st: 0%, 2nd: 10%, 3rd: 10%	1st: 27%, 2nd: 42%, 3rd: 72%
Position of the injectors used	# 2,4,7,8,10,12,14,16,17, 18,19,20,22,24,26,27,29,31	# 8,9,10,12,13,14,15,16,18, 19,20,21,22,23,25,26,27,28
% pressurisation (input)	100 %	63 %
% extractor	90 %	61-64 %
Suspension injection pressure	20 bar	21-22 bar
Depression	-5 mm H <sub>2</sub> O	-6 to -8 mm H <sub>2</sub> O
Sticking emission	323 kg/h	27 kg/h



Sum of diameters: 58.1 mm; Sum of areas: 140.3 mm<sup>2</sup>

Figure 13. Service conditions (final adjustments), surface map of wall sticking.

### 7. CHARACTERISATION OF ADHESION IN THE LABORATORY

The figure 14a shows that the residual moistures of the raw materials were quite similar to each other, with the exception of kaolin 8, possibly due to its much higher viscosity than the other mixtures: the obtainment of a same mean drop diameter required reducing the liquid flow rate, which is why the drying air in flight presented a greater relative efficiency. Even with little residual moisture, this kaolin displays, like kaolin K, a higher deposition efficiency than the illitic mixtures A and D. Since all the slurries were fully deflocculated and the sulphate concentrations did not reflect a clear tendency in this respect, it would be convenient to study the role of particle size. The correlation proposal in figure 14b suggests the importance of the particle fraction finer than 0.4  $\mu\text{m}$  in the kaolins used, probably due to its greater capacity to generate fine capillaries and a greater specific adhesion force by liquid bridges<sup>[6]</sup>. Note that preheating of the substrate gives rise to a slightly diminished, albeit still significant, efficiency.

The measurement of the material lost by centrifugation provided similar results under tension (figure 15a) and shear (figure 15b) for the deposits on cold substrates. The first layers resisted the applied stresses very well, but their mechanical strength decreased after the second spraying. There was little difference in behaviour between clay A by itself and mixed with non-plastics (D). In spite of the similarity between the tension and shear results for cold substrates, the deposits on preheated substrates (DD) were much less resistant to shear than tension, particularly in the case of the first layers. This greater initial facility of detachment by shear could explain the poor adhesion observed in the vertical walls of the spray dryers, when the walls are correctly preheated (spray dryer start-up projecting water in the injectors to initiate the cycle, instead of initiating it directly with slurry). If, on the contrary, the pulverisation of the clayey drops begins when the spray dryer has not yet reached suitable temperatures, the beneficial effect of preheating could be less relevant.

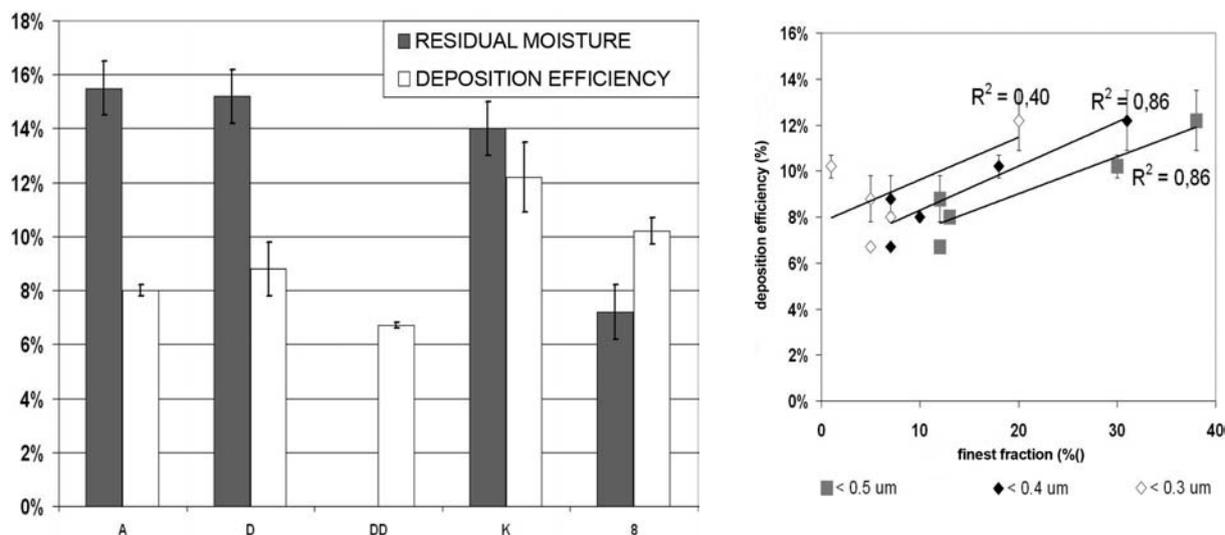


Figure 14. Characteristics of the adhered layers:

(a) moisture and deposition efficiency; (b) correlation between efficiency and fine particle fraction.

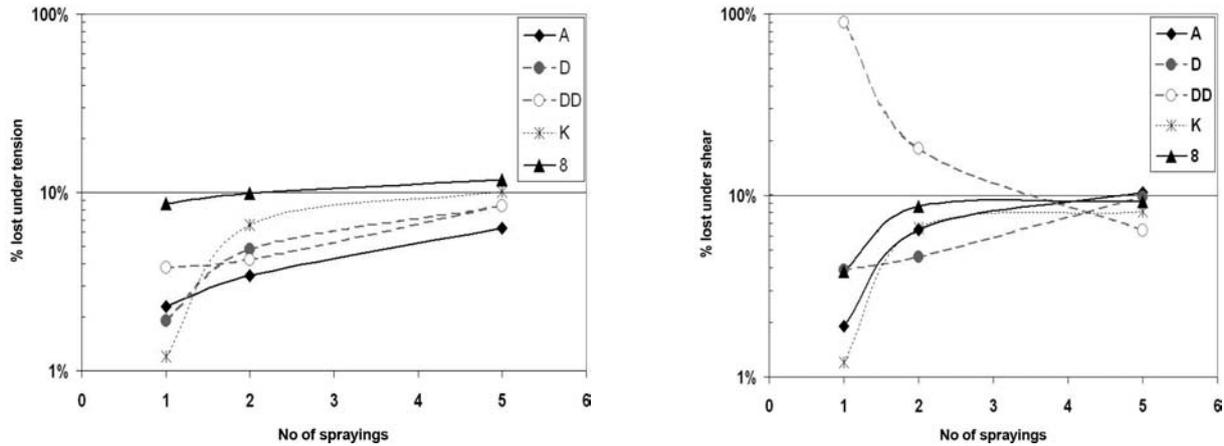


Figure 15. Behaviour in centrifugation of the layers: (a) under tension; (b) under shear.

## 8. CONCLUSION

The observations made in plant enable classifying the various types of wall sticking in industrial spray dryers. By implementing a crown plan matching the arrangement and correct opening of the drying air distribution windows, and the formation of an appropriate depression in the extractor, the sticking emissions were reduced (kg/h) by 92%, and the covered wall surface decreased from 60% to 5%. Note that the design of the main air windows forces the user to avoid placing injectors in certain sectors of the crown, and to try to distribute equitably the drying air in the tower. The usefulness of the mass and mapping methods as tools deserves to be noted for measuring this undesirable phenomenon on an industrial scale. These results demonstrate the possibility, for the person in charge of the body preparation department to maintain high production with little wall sticking, with certain ease, working on the basis of the factor 'handling of the spray dryer'. On the other hand, the factor 'raw material' evidenced some effects, notably regarding the importance of the submicron fraction (in particular, below  $0.4 \mu\text{m}$ ) in clays. Simple, novel measurement tools have been proposed to study the proneness to wall sticking by adhesion (centrifugation in the 'shear' mode) and the deposition efficiency of the projected drops, which can be extremely useful when it comes to designing new mixtures. Furthermore, these methods highlight the role of substrate temperature and suggest the suitability of adequate preheating of the vertical walls of the spray dryer to limit the formation of layers on an industrial level.

## 9. ACKNOWLEDGEMENTS

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