Characterization of Flow Properties of Powder Coatings Used in the Automotive Industry[†]

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Abstract

The aim of this work was, on the one hand, to gain a better understanding of the effect of flow additive content on the powder flowability, and on the other hand, to point out the most suitable tests to characterize the flow properties of industrial powder paints used in automotive industries. The flow properties of 5 powder coatings, containing 0, 0.12, 0.30, 0.53 and 0.96 w/w%, respectively, of a flow additive and an industrial batch, were tested using both conventional and novel characterization techniques. The lubricant used was a silica powder. Test methods employed were a packing test, a circular shear cell (Peschl), a powder rheometer and a fluidization/de-aeration test. The flowability of powder batches is significantly improved with increasing lubricant content up to an optimal value of about 0.53%. SEM images of different powder samples showed that the optimal point corresponds to a critical additive content where the amount of additive is high enough to form a continuous film around the particles. Beyond this critical content, the particle-lubricant contacts are replaced by lubricant-lubricant contacts. This phenomenon leads to a degradation of flowability due to a higher cohesivity of additive particles.

INTRODUCTION

Among all manufacturing processes for automotive production, the painting operation contributes most to direct environmental emissions. As a consequence of recent restrictions in European legislation concerning the volatile organic compounds (VOC) emissions, the trend in almost every finisher industrial field is to replace the conventional solvent-borne paints by new low-emission paint systems, including powder coating systems. Powder paints are very finely divided solvent-free polymer coatings, which present important advantages over conventional paints from ecological and economical points of view [1].

There are many ways to apply powder coating materials, as reported in the “technical” literature.

The most important ones are without doubt the fluidized bed technique and the electrostatic spraying of powders.

The fluidized bed was the first application method used to apply powder coatings. Powder paint is suspended inside a fluidized reservoir by blowing air through it from a porous base. Workpieces, preheated to a temperature above the melting point of the powder, are then immersed in the fluidized powder, and powder particles in contact with the substrate melt and adhere to the surface. This technique is still used in many applications such as wire products or electrical busbars. However, the development of this process has been limited by large drawbacks: the relatively high thickness of the final layer on the one hand, and the impossibility of handling on the other hand.

Compared to fluidized bed coating, the powder electrostatic spraying technique is much more versatile and can be applied to a wide variety of workpieces with different shapes and sizes. Furthermore, this technique provides thinner and more homogeneous

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films.

These advantages explain in part the great interest shown in this technique among industrial finishers and more particularly the automotive sector. Since the introduction of this technique in 1962, the electrostatic powder spray process outstripped the fluidized bed technique.

As shown schematically in **Figure 1**, in electrostatic powder coating processes, powder paint is fluidized in a reservoir and is then blown through a feed pipe to a special charging corona bell. After passing through the corona bell, in which particles are electrostatically charged, the powder is sprayed toward a grounded workpiece [2]. The adhered powder is then heated, whereupon it melts and cross-links to form a uniform layer over the workpiece. In addition, unlike the liquid paint systems in which non-deposited paint is lost, the oversprayed powder during the electrostatic application process can be reclaimed for further use.

Fig. 1 Schematic diagram of industrial application process

In the automotive industry, a very high-grade appearance is required for a minimal thickness of finished film. However, powder coatings commonly suffer from the so-called “orange peel” effect, a subtle surface unevenness of the solidified coating, which influences the final appearance. In the case of powder coatings, the finished film quality can be reached by very specific physical properties of powder: small and narrow particle size distributions [3]. Unfortunately, as is well-known in powder technology, the handling properties of these fine powders present some problems such as blocking during storage and poor powder flowability that cause application problems such as poor fluidization, irregular feed rates and increased powder hold-up in the transport hose [4]. In powder

technology, one of the most common solutions to overcome this problem is to add a flow additive: particles of submicron size located between large particles, resulting in a reduction of inter-particle forces [5].

The aim of this work was, on the one hand, a better understanding of the effect of flow additive content on the powders’ flowability and, on the other hand, to point out the most suitable tests to characterize the flow properties of industrial powder coatings used in automotive industries. The flow properties of 5 powder coatings, containing 0, 0.12, 0.30, 0.53 and 0.96 w/w% of a flow additive and an industrial batch, respectively, were tested using current and novel characterization techniques. The lubricant used was a silica powder. Test methods employed were the packing test, circular shear cell (Peschl), powder rheometer and fluidization/de-aeration tests.

EXPERIMENTAL

1. Materials

A powder based on polyester/epoxy thermosetting hybrid resin was chosen for this study. It is a gray powder coating used for the primer coat in the automotive industry. It has an angular particle shape. The mean particle size is close to $29\text{ }\mu\text{m}$ and the true density 1310 kg/m^3 . According to Geldart’s classification, the original powder, which does not contain the flow additive, falls within group C [6]. Powders that are in any way cohesive belong in this category. Fluidization of such powders is extremely difficult and they flow poorly.

The additive used is a commercial, very fine spherical silica powder (10 nm in diameter). This kind of additive is frequently used in order to improve the flowability of cohesive powders. A silica component was added to the fresh powder in weight percentages of 0; 0.25; 0.50; 1.00 and 2.00%. Each powder mixture was prepared in two steps inside a fluidization column. A first blend is carried out in a 1.10^{-2} m^3 fluidization column in order to pre-dilute the flow additive in 4 kg of fresh powder coating. The mixing time was 4 hours under mild fluidization. Then, the mixture was added again to 16 kg of fresh powder paint into a 1.10^{-1} m^3 stainless steel column. The mixing time was then 12 hours. The final batch weight was therefore 20 kg. During fluidization, oil-free air dried through a bed of silica gel was used. The use of a mechanical stirrer improved the fluidization and enabled the use of a low superficial air velocity close to $2.10^{-3}\text{ m.s}^{-1}$ in order

Table 1 Characteristics of powder paints

Product	AGF 1	AGF 2	AGF 3	AGF 4	AGF 5	Industrial Batch
Additive content, %w/w, (based on added amount)	0	0.25	0.50	1.00	2.00	–
Additive content, %w/w, (measured)	0	0.12	0.30	0.53	0.96	–
Mean diameter, d_v (μm)	29.2	–	–	–	–	28.8
Mean diameter, d_{sv} (μm)	21.6	–	–	–	–	21.2
Shape factor	0,8	0,8	0,8	0,8	0,8	–
True density (kg.m^{-3})	1310	1310	1310	1310	1310	1310
Blaine specific area ($\text{m}^2.\text{kg}^{-1}$)	2501	2531	2656	2766	3386	2448

to limit the loss of fine particles. Finally, each batch was sieved through a 75- μm mesh. In this work, an industrial batch of powder primer was also studied in order to assess the suitability of the test for industrial use.

The six powder coatings containing different amounts of additives were fully physically characterized. The particle size distribution was measured by laser diffraction using a Malvern Mastersizer (manufactured by Malvern Instruments), and the true density of solids by a Helium Pycnometer Accupyc 1330 (manufactured by Micromeritics). The true percentage of flowability additive contents in the final batches was also determined using the ICP-AES method [7]. The specific surface area was assessed using a Blaine permeameter.

Table 1 summarizes some physical characteristics of the 6 batches used in this work.

2. Methods

2.1. Aerated and tap density measurements

The bulk density of a powder is its mass divided by the bulk volume it occupies. The value of bulk density depends tremendously on the consolidation state of the powder. So both the aerated and tap densities of a powder have very different values [8,9].

Measurement of the bulk volume variation of the powder during tapping was performed using a tap density volumeter (manufactured by Quantachrome). It consists of a graduated 100-ml vessel and a tapping apparatus. The simple experimental procedure was the following: the powder was first poured into the 10^{-4} m^3 graduate cylinder through a suitable vibrating sieve such that most of the material would pass readily. The vibration amplitude was such that the time necessary to fill the vessel was at least 10 s. Before

weighing, the excess powder was scraped from the top of the vessel using a ruler, without disturbing or compacting the loosely settled powder. The vessel was then subjected to successive vertical taps, and volume measurements were obtained after a different number of taps. For each sample, the operation was continued up to 2000 taps, whereupon a steady state was reached indicating that the packing was achieved. The bulk density of a powder, ρ_N , at each tapping number N was determined by dividing the mass of the sample by the volume it occupies. Three tests were conducted on each sample for aerated density and tapped density. The average values were taken to be the aerated and tapped bulk density. The powder volume decrease was assessed by several parameters.

The first one is the ratio of aerated and tap density, known as the Hausner Ratio, HR, calculated using the following equation:

$$HR = \frac{\rho_N}{\rho_O} = \frac{V_O}{V_N} \quad (1)$$

where ρ_O is the bulk density of unpacked powder, V_O the initial apparent volume and V_N the powder volume at tapping number N . Typically, a powder that is difficult to fluidize will have an HR greater than 1.4. A powder that exhibits excellent flow and fluidization will be around 1.25 or less.

The second parameter is the compressibility C (introduced by Carr [10]) of the powder, which is the degree of volume reduction given by:

$$C = \frac{V_O - V_N}{V_O} \quad (2)$$

Both Hausner's ratio and compressibility are commonly used as qualitative indicators for determining whether or not a powder is cohesive. They reflect the friction conditions in a moving mass of powder rather

Table 2 Carr's classification

$0.05 < C < 0.15$	good flow
$0.15 < C < 0.18$	fair flow
$0.18 < C < 0.22$	passable flow
$0.22 < C < 0.35$	poor flow
$0.35 < C < 0.40$	very poor flow

than in a static situation. Generally, the structure of a cohesive powder will collapse significantly on tapping while the free-flowing powder has little scope for further consolidation. The powder particles are forced to jump and to lose contact with each other for a moment while tapping. When the friction between the particles is reduced, the particles rearrange, and thus tapping results in improved packing conditions. So, a drop in Hausner ratio or compressibility corresponds to a decrease in the cohesiveness of the powder [8].

Concerning the compressibility C , the simplified Kawakita and Lüdde equation [11] leads to a relationship between the tapping compaction of powders and their compression:

$$\frac{N}{C} = \frac{1}{a} \cdot N + \frac{1}{ab} \quad (3)$$

a and b are constants, characteristic of the powder. The linear relationship between N/C and N allows the constants to be evaluated graphically.

The constant a stands for the degree of volume reduction at the limit of tapping and represents the maximal compressibility and the fluidity of a powder [12].

Conversely, b represents the tapping compressibility of a material. Its reciprocal $1/b$ is a measure of the resistance of the material to tapping. Hence, $1/b$ is considered as the constant relating to the cohesive forces of powder particles [12]. In this work, $1/b$ has not been taken into account because a specific experimental method is necessary to obtain an accurate value.

2.2. Shear cell measurement

Yield locus and failure functions of the six powders under investigation were established using a Peschl circular shear test (RO-200, IPT), **Figure 2**. The device consists of a 60-mm rotating bottom ring with a roughened base, an open upper ring of equal diameter and a round roughened lid prolonged by a horizontal arm permitting measurement of the shear force during experiments [13].

Firstly, the sample was compressed for 45 minutes under predetermined consolidation pressure (16, 12, 8; 4 and 2 kPa) before performing the shear test. Commonly, this step is called the “preconsolidation step”. In order to compensate the volume decrease of the consolidated powder, an extension ring is used to permit the addition of extra powder. When the consolidation time is completed, the extension ring is removed and the powder is carefully scraped level. The shear cell is then set up over the rotational base.

The rotational base is then activated in the direction of the shear. When the horizontal arm contacts the force sensor, the lid motion is stopped. Consequently, by rotating the shear cell relative to the lid, a shear deformation is developed leading to a shear stress, acting in the assumed shear plane located between the upper ring and bottom ring. Generally, the shear stress increases until failure takes place and then remains constant.

The first shear step is a conditioning step, which has the purpose of bringing the sample to critical conditions (shear without change of volume). We can call this step the “consolidation step”. This consolidation step has to be repeated by using a constant vertical load until the maximum top stress value is reached. After reaching the steady-state shear stress, the sample is relieved of the shear stress by reversing the direction of the rotating base until the shear stress becomes zero.

Fig. 2 Schematic diagram of circular shear stress apparatus (Peschl)

In the period where the shear stress is equal to zero, the vertical load is changed in order to prepare

the sample for the measurement in the next step. A shearing test which includes 10 steps starts with the same vertical load as the preconsolidation step and continues decreasing the normal load step by step. So the shear stress (τ), necessary to cause failure and create flow under 10 applied normal stresses (σ), can be measured for a given consolidation. The curve obtained is the so-called yield locus. Two further important parameters may be extracted from these results:

- Unconfined yield strength (f_c): this parameter is obtained by plotting the Mohr semicircle which passes through the origin and is at a tangent to the yield locus.
- Major principal stress (σ_I): it is obtained by a Mohr semicircle which is at a tangent to the yield locus at its end point.

According to Jenike [14], the ratio of the major principal stress σ_I at steady-state flow to the unconfined yield strength f_c called the flowability index is a good indicator of powder flowability under load:

$$i = \sigma_I / f_c \quad (4)$$

Table 3 Classification of flowability index

$i < 2$	very cohesive
$2 < i < 4$	cohesive
$4 < i < 10$	easy flowing
$10 < i$	free flowing

In this work, five yield loci (2, 4, 8, 12 and 16 kPa) were established for each sample from which the corresponding value of f_c and σ_I were determined. A curve of (f_c) vs (σ_I), known as the flow function (FF), is then plotted and the flowability index determined.

2.3. Powder rheometer measurement:

The FT4 powder rheometer (manufactured by Freeman Technology) is a new device that is able to classify powders regarding their flowability. The aim of this device is to provide an automated testing program that is relatively independent of the operator and is quick.

The device principle, which is represented in **Figure 3**, is simple [15]. A powder sample, after being weighed, is placed in a glass cylindrical vessel. A specific twisted blade, with a diameter of 60 mm, moves along a helical path through the powder column. The controlled parameters are helical path angles, blade

tip speed, starting height and final height. As the parameter values are changed, the helical path has more or less revolution and the blade moves downward or upward, in clockwise or anti-clockwise direction. Therefore, it is possible to choose the direction of the blade tip displacement. Different regimes are possible: a shearing regime (the blade tip is parallel to the trajectory) or a compaction regime (the blade tip is perpendicular to the trajectory). The blade motion imposes the forces, causing the deformation and the flow of the powder. The axial forces and rotational forces acting on the blade during the cycle through the powder are measured continuously and used to derive the work done, or energy consumed, in displacing the powder. A typical test program alternates two steps. The first step is preparation of the sample for testing in a conditioning process in which the blade gently displaces the powder to establish a consistent and reproducible packing density. During the second step of the test cycle, the blade moved along a downward helical path, but in the opposite direction, to impose a compaction regime, thereby forcing the powder to flow around the blade.

Fig. 3 Schematic diagram of FT4 powder rheometer from Freeman Technology

In this study, a specific test called the aeration test was developed. The bottom of the cylindrical test vessel is made of a stainless steel porous plate 62 mm in diameter. During the test, an air flow controlled by a mass flow controller passes from the bottom to the top through the column of powder. Dependent on the air velocity, the powder bed is either fluidized or sim-

ply aerated.

Approximately 100 g of powder paint is loaded into the translucent vessel. For the same sample, the measurements were achieved at 5 superficial air velocities (1, 2, 4, 6, 8 mm/s). A complete test is carried out with the air flow varying from the highest to the lowest value. In between each new air velocity, the testing stage is preceded by a conditioning cycle in order to obtain a steady state of aerated powder. During the aeration test, the normal force developed by the blade is very low. So for this study, only the torque energy was analysed.

2.4. Fluidization/De-aeration measurement:

A fluidization test and a bed collapsing technique were used in order to assess the fluidization and the cohesiveness of the six powders and to classify them. Virtually, the structural or inter-particle forces that affect flowability also affect fluidization.

Fig. 4 Schematic diagram of experimental apparatus

The equipment to assess fluidization behavior in this study consisted of a rather standard fluidized test column. Three main parts comprise this experimental apparatus: the column, the air flow circuit and the acquisition device. **Figure 4** shows a schematic diagram with the dimensions of the experimental appara-

tus. The trials were carried out in a Pyrex column 100 mm in diameter and 500 mm in height built on an aluminum frame to make transport of the apparatus easier. At the top of the column, an expanded disengaging section minimizes particle entrainment and a cover surmounted by a cyclone recovers the finest particles. The distributor was made of two overlapping filter papers supported by a metallic porous plate. It is supported by the windbox, which was made deliberately small ($7,85 \cdot 10^{-4} \text{ m}^3$) in order to reduce the escape time and resistance during the de-aeration tests. Compressed air was fed to the bed of powder through a filter, a pressure regulator, a computerized mass flowmeter regulator (4), a solenoid valve (3), the windbox and the distributor plate. Transducers continually recorded the relative humidity (5% HR) and the temperature (22°C) in the windbox. During the tests, the overall pressure drop of the powder bed was measured by a relative pressure transducer (Kobold 3277-BO15) connected to a pressure tap at the windbox.

By means of an acquisition system, a program was created to increase and decrease the air flow rate for a certain time period. So, fluidization trials were carried out more than three times to automatically determine the minimum fluidization velocity for the different tested powders.

The powders' cohesiveness can also be quantified by the de-aeration method. For example, it is usual to foresee the ability of a powder to be conveyed in a pneumatic dense phase or lean phase by a de-aeration test. Initially, a known weight of solids (750 g) was filled into the column. The powder was then fluidized by compressed air at 2 times U_{mf} . When the system reached steady state, the air supply was suddenly cut off by a solenoid valve and the collapse height was recorded as a function of time using a device comprising a float and a laser beam. With the float system, the rapidly collapsing bed height could be determined with "reasonable" accuracy. The residual gas in the windbox was purged to the atmosphere through a solenoid valve (2) and a needle valve (1) installed on the windbox [16]. Without this device, after the sudden cut-off of the air supply, the residual gas in the windbox will be purged through the bed of particles and will disturb its collapsing. Thanks to an optimum valve opening factor, this phenomenon is limited. Because the bed height at $t < 0$ fluctuates considerably due to bubbling, up to 5 repeat tests were made and a numerical average taken which was then plotted. Collapse tests were done with the six powders over a range of velocities above U_{mf} .

RESULTS AND DISCUSSION

1. SEM

By using Scanning Electronic Microscopy (SEM), the surface of a powder coating host particle can be

explored in order to look at the surface coverage by submicronic silica particles.

The micrograph in **Figure 5** gives an overview of paint particles. The angular shape is the shape of ground particles. The micrographs in **Figures 6 to 10** compare the surface coverage for various amounts of

Fig. 5 Powder paint particles

Fig. 6 Particle surface without flow additive

Fig. 7 Particle surface with 0.12% of flow additive

Fig. 8 Particle surface with 0.30% of flow additive

Fig. 9 Particle surface with 0.53% of flow additive

Fig. 10 Particle surface with 0.96% of flow additive

added additives. The nature of the attached particles was checked by the X-ray probe of the SEM.

2. Packing measurements

A greater inter-particle porosity indicates the presence of more air entrapped between the particles and consequently a high compressibility, which corresponds to a cohesive powder and thus poor flowability. In contrast, a low compressibility denotes lower cohesiveness.

A drop in the Hausner ratio or in the compressibility C involves a decrease in cohesiveness of the powder [8]. According to Carr's classification [9,10], the results presented in **Table 4** and in **Figure 11** highlight the flow property improvement when the flow additive percentage increases. *AGF 1* is therefore classified as having poor flow whereas *AGF 4* has a good flow. Nevertheless, it is interesting to see that the flowability improvement reaches a maximum. Beyond an optimal amount of silica, the flowability of powder drops again. This is indicated by the deterioration of *AGF 5* flow properties. The *AGF 5* flowabil-

ity hovers between *AGF 1* and *AGF 2*. This result also shows the important cohesion of the industrial batch which has a compressibility close to *AGF 1*.

3. Shear cell measurements

Figure 12 shows the results obtained using the circular shear cell for the unlubricated powder in comparison with mixtures containing silica. The best flow properties are obtained for a silica concentration which gives the highest value of flowability index (i).

As expected, the increase of the flow additive percentage from 0% (*AGF 1*) to 0.53% (*AGF 4*) improves the flow properties of the powder paint, since the flow function FF moves to the "easy-flowing" area. These results show also that above 0.96% of flow additive (*AGF 5*), the flowability drops. The *AGF 5* flow properties are very similar to *AGF 3*. However, the effect of lubrication appears less clearly than with the flowability index. This technique is less suitable and less sensitive for characterizing the powders. This result also shows the important cohesiveness of the powder of the industrial batch.

Table 4 Packing test results

Powder	AGF 1	AGF 2	AGF 3	AGF 4	AGF 5	Industrial Batch
a (Kawakita and Lüdde)	0.244	0.165	0.148	0.122	0.190	0.210
RH	1.32	1.20	1.18	1.14	1.23	1.26
C (%)	24	16.3	14.7	12.0	18.7	20.8
Carr's Classification	Poor flow	Fair flow	Fair flow	Good flow	Passable flow	Passable flow

Fig. 11 Relation between N/C and the number of taps N

Fig. 12 Flow function and classification according to the flowability index (i)

4. Powder rheometer measurements

In **Figure 13**, results show the total torque energy consumed during a downward test traverse as a function of the superficial air velocity through the distributor. Raw data recorded between a height of 60 and 20 millimeters from the bottom of the vessel were used for the analysis.

Generally speaking, for the highest air velocities, an easy-flowing powder leads to homogeneous fluidization and the torque energy measured is very low. In contrast, fluidization is very difficult for a cohesive

powder and leads to channeling of the powder bed. The torque energy value is increased. When the gas velocity is decreased, the powder bed goes from the fluidized state to the consolidate state and the energy increases. These plots show that the torque energy decreases whereas the flow additive percentage increases up until 0.53% (*AGF 4*). This test did not show differences between *AGF 5*, *AGF 4* and *AGF 3*. The very low torque energy values of these three blends reveal good fluidization properties. However, at a low air velocity (1 mm.s^{-1}), the flow properties of *AGF 5* are slightly lower than *AGF 3*. These results

Fig. 13 Energy as a function of aeration air velocity

show that the powder paint without lubricant (*AGF 1*) is very cohesive. For each air flow rate, the torque energy values are much greater than for other mixtures with silica. Regarding the torque energy value at an air velocity of 1 mm.s^{-1} , *AGF 5* takes its place between *AGF 2* and *AGF 3*. In addition, these results also show the stronger cohesiveness of industrial powders. The very low energy values recorded at higher air velocities indicate that fluidization of this powder is possible. Thus, this method seems suitable for industrial application.

5. Fluidization/De-aeration measurements

In a first stage, the minimum fluidization velocities U_{mf} of the six powder coatings were measured experimentally (**Table 5**). No measurements could be obtained for the powder *AGF 1*, (without flow additives), because fluidization was not possible. It denotes the very cohesive nature of this powder.

Table 5 Fluidization results

	Experimental U_{mf} (mm/s)
AGF 1	—
AGF 2	2.82
AGF 3	1.06
AGF 4	1.06
AGF 5	1.12
Industrial Batch	3.67

The other powders provide very different values of U_{mf} . The *AGF 2* powder (0.12% of additive) displays an U_{mf} nearly 3 times greater than the powders with higher additive content. Materials that exhibited good flow characteristics (0.30, 0.53 and 0.96%) provide a U_{mf} value close to 1 mm.s^{-1} and are difficult to differentiate, although the flow additive quantity increased. Moreover, in comparison with the other flowability test results, the *AGF 5* powder did not display a significant gap of behavior with regard to the other powders.

The powder from the industrial batch deviated clearly from the other powders with a more significant U_{mf} value. It shows the very cohesive nature of the powder coating used by the automotive industry. However, the fluidization of such a powder improved by the use of vibrations is quite suitable in the industrial application process.

In a second phase, de-aeration tests were undertaken. The total times for the de-aeration process and the total heights of initial expansion recorded for a superficial air velocity of 2 times U_{mf} are listed in **Table 6**. The curves in **Figure 14** display the kinetics of de-aeration of five of the six powders. The *AGF 1* powder, which did not contain a flow additive, is not fluidizable and has thus not been studied. The results show different behavior among the powders.

Regarding the curves in **Fig. 14**, 2 groups of de-aeration rate are observed. When fluidized at 2 times U_{mf} , the *AGF 2* powder and the industrial batch present a greater initial expansion, and the presence of channeling is visible particularly for the industrial

Fig. 14 Bed collapse curves

batch. When the air is cut off suddenly, they de-aerate more rapidly than the other powders, as the air escapes through the channels formed within the bed of powder. This affects the rate of de-aeration as shown in **Fig. 14**. The collapse curves are close to those of the cohesive group-C powders of the Geldart classification [17]. Powders which are intrinsically more cohesive de-aerate quickly because of channeling. After an initial rapid collapse of the bed caused by cracks caving in, the rate of collapse is controlled by the rate at which air can escape to vertical channels formed in the bulk of the powder. Regarding the *AGF 3*, *AGF 4* and *AGF 5* powders no channeling was observed, but the bubbling near the surface of the bed was more pronounced. Besides this, after the air was shut off, the collapsing of the powder bed is slower and the kinetic of de-aeration recorded is close to one of the group-A powders of the Geldart classification, which presents the height decreasing almost linearly [17].

Table 6 Collapsing results

	Total Expansion (mm)	Time (s)
AGF 1	–	–
AGF 2	85	20
AGF 3	49	22
AGF 4	52	26
AGF 5	58	27
Industrial Batch	69	18

The initial expansion height of the different powders did not provide a direct classification correlated to their flowability. In contrast, the total time for the de-aeration process leads to a classification directly correlated to both the flowability and fluidization properties of the six powders as indicated by the different characterization techniques. However, the deterioration of the flow properties of the *AGF 5* powder found by other methods was not observed. The fluidization properties of *AGF 5* appear very close to that of *AGF 4*.

CONCLUSION

Different methods were used to investigate the effect of flow additive content on the flow properties of powder paint. The addition of silica was followed by particle size and permeability measurements, as well as SEM analysis. The accurate quantification of silica

contents was achieved by ICP-AES.

Not every test showed the same sensitivity. Meaning that the classification of the powders according to their flowability or fluidization is slightly different depending on the method used. Moreover, not all of these techniques are suitable for use as a simple bench-top test as required by industry.

The tapping test, shearing test, powder rheometer, fluidization and de-aeration tests show as expected [18-19] that the flowability improves with the lubricant content up to a percentage of 0.30%.

Beyond 0.30%, the tests with some consolidation (tapping test and shearing test) show that an optimum in additive concentration exists, corresponding to the amount suitable to cover the whole particle surface. The tapping test shows greater resolution, is less time-consuming and less tedious.

Fluidization/de-aeration and powder rheometer tests are discriminating only for poorly fluidizable powders, which is the case for industrial powder paints where usually the flow additive content added is rather low in order not to adversely affect other properties. These tests are fully automatic and relatively fast, which is a plus for an industrial bench-top test.

LIST OF SYMBOLS

a	constant of Kawakita and Lüdde equation	
C	compressibility of a powder	
$d_{4,3}$	particle size diameter	(m)
$d_{3,2}$	particle size diameter	(m)
f_C	unconfined yield stress	(Pa)
FF	flow function	
H	height of bed at any time t	(m)
H_O	height of settled bed of powder	(m)
HR	Hausner Ratio	
i	flowability index	
N	tapping number	
t	time	(s)
U_{mf}	minimum fluidization velocity	(m·s ⁻¹)
V_N	powder volume at tapping number N	(m ³)
V_O	initial apparent powder volume	(m ³)
ρ_N	bulk density of a powder at each tapping number	(kg·m ⁻³)
ρ_O	initial bulk density of a powder	(kg·m ⁻³)
σ_I	major principal stress	(Pa)

REFERENCES

- [1] Lovano S.: Automotive applications for powder coating, Metal Finishing, (1996), 20-24
- [2] Bailey A.: The science and technology of electrostatic

- powder spraying, transport and coating, *Journal of Electrostatic*, 45 (1998), 85-120
- [3] Zhisong H., Scriven L.E., Davis H.T.: Film formation in powder coatings, *Waterborne Higher solids and powder coatings symposium*, (1997)
 - [4] Satoh H., Harada Y., Libke S.: Spherical particle for automotive powder coatings, *Progress In Organic Coatings*, 34 (1998), 193-199
 - [5] Fluck D., Fultz J., Darsillo M.: Fluidization additives in powder coatings, *Paint & Coatings Industry*, (1998), 214-220
 - [6] Geldart D.: Type of gas fluidization, *Powder Technology*, 7 (1973), 285-292
 - [7] Hermmelin M., Mermet J.M., Bertucci M., Zydowicz P.: Determination of additives in PVC material by UV laser ablation inductively coupled plasma atomic emission spectrometry, *Spectrochimica Acta*, B52 (1997), 421-430
 - [8] Abdullah A.C., Geldart A.: The use of bulk density measurements as flowability indicators, *Powder Technology*, 102 (1999), 151-165
 - [9] Viana M., Gabaude-Renou M.D.C., Pontier C., Chulia D.: The packing coefficient: a suitable parameter to assess the flow properties of powders, *KONA*, 19 (2001), 85-93
 - [10] Carr R.: Evaluating flow properties of solids, *Chemical Engineering*, 18 (1965), 163-168
 - [11] Kawakita K., Lüdde K-H: Some consideration on powder Compression Equations, *Powder technology*, 11 (1970), 61-68
 - [12] Yamashiro M., Yuasa Y: An experimental study on the relationship between compressibility, fluidity and cohesion of powder solids at small tapping numbers, *Powder technology*, 34 (1983), 225-231
 - [13] Peschl I.A.S.Z.: Measurement and evaluation of mechanical properties of powders, *Powder handling & processing*, 1 (1989), 135-141
 - [14] Schwedes J.: Testers for measuring flow properties of particulate solids, *Powder handling & processing*, 12 n° 4 (2000), 337-354
 - [15] Freeman R.: The classification of powders in relation to flowability, *Powder Handling and Processing*, 12 n° 2 (2000), 194-197
 - [16] Park J.J., Park J.H., Chang I.S.: A new bed-collapsing technique for measuring the dense phase properties of gaz-fluidised beds, *Powder technology*, 66 (1991), 249-257
 - [17] Geldart D., Wong A.C.Y.: Fluidization of powder showing degrees of cohesiveness – II. Experiments on rates of de-aeration, *Chemical Engineering Science*, 40 (1985), 653-661
 - [18] Podczec F., Miah Y.: The influence of particle size and shape on the angle of internal friction and the flow factor of unlubricated and lubricated powders, *international journal of pharmaceuticals*, 144 (1996), 187-194
 - [19] Feliw G.: *Ecoulement de milieux granulaires en tambour tournant*, Thèse de doctorat, Institut National Polytechnique de Lorraine – Nancy, (2002)

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