An investigation of coat growth rate and surface coverage in coating of particulars.

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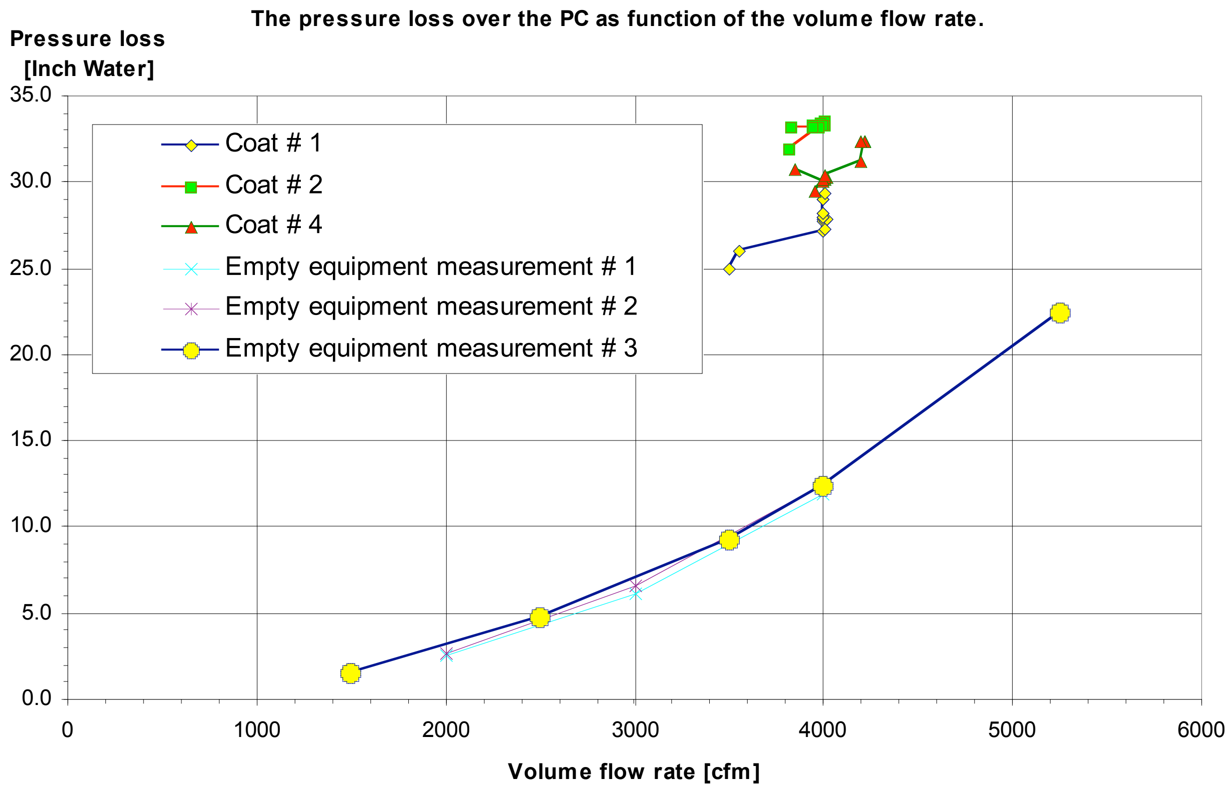
Introduction.
When coating particulars, several physical elements have to be taken into consideration, like coating yield, attrition, agglomeration, coat quality and stability of the process.
The product to be coated has its own characteristics, which, together with the physical peculiarities of the coat, makes it clear that the design of the coating equipment has to be flexible enough to accommodate these variations. When undesirable effects occur, such as a low coating yield, extensive attrition or the appearance of large agglomeration fractions, it is important to focus on what is actually happening inside the equipment and find out how to change the process to avoid those unwanted effects.

Attrition.
Primary particulars - particulars before coating - are more friable than coated particulars. To diminish the loss due to attrition, the solution is to spray faster, closer to the saturated condition, and to slow down the process gas flow in the initial phase, to spare the primary particulars. The search for a solution enabling to coat closer to the saturated condition led to what is called Real Time Process Determination. The other way to battle unwanted attrition was to focus on a way to find out how long it would take to cover the surface of any size particulars with just one layer of coating, making them less friable in the process. The next step is to find a method to determine the product flux of particulars through the equipment and control this flux.

Coverage of the surface of particulars and coat growth rates.
One of the first estimates of coating surface flux was done on a 400 [□ m] diameter particular. The calculation showed that the coat growth rate was 2.5 [□ m/h]. Normally, the thickness of the polymers, of which a coat is made, ranges from 0.33 [□ m], or in nanometer 330 [n m], to close to 400 [n m]. The result was surprising: it showed that per hour only six to eight layers of coating were applied. If each single coating application is three or four polymer layers thick, each particular would be coated only two times per hour. Measuring the velocity of the product in the down flow bed through the sight glass, we concluded that each particular would pass through the spray zone with an interval of a few minutes, meaning that a particular had to circulate 20 - 30 times to be coated once. Experiments showed that the Precision Coater, with swirl generator in the inlet, achieved the full coverage of the particulars at a coat thickness of 1.5 [□ m]. Without swirl, full coverage of the particulars was first accomplished at a coat thickness of 2.5 [□ m]. The conclusion of these experiments was that it was impossible to apply coating to the particulars as a monolayer (one polymer thick), because the physical behaviour of the coat forced it to be minimal four to six polymer layers thick.
Next problem was to find out how much of the surface area of the particulars would be covered each time they passed through the spray zone, assuming that the difference in pressure loss between the empty equipment and the pressure loss, caused by the equipment in process mode, must be the work to move the product through the equipment.

Method to determine the particular's surfaces flux through the spray zone.
To determine the number of particulars passing through the spray zone, we have to combine the local with the global calculation. We use the conservation of mass and the first law of thermodynamics for a control volume with flow over the volume boundary. The control volume is established around a single particular. We assume that there is no change of mass inside the control volume in time. This is not totally correct; we applied coat and solvent to the particular. But we assume this has no influence on the change of the particulars' velocity or path. We assume that the work done on the particular, transporting it through the equipment, is the pressure loss inside the control volume. The enthalpy for the droplet hitting the particular, the heat applied to the particular to evaporate the solvent and the vapour flowing over the control volume's boundary, are not affecting the pressure loss inside the control volume. We say that the droplets hit the particular equally from all sides, so there is no gain or loss of work to the particular from the impact of the droplets.
If we measure the pressure loss in the equipment when no particular is present, we know the process gas loss. When we place the particulars in the equipment, we observe that the pressure loss increases. This must be due to the particulars, which we transport around in the equipment.



This is the global pressure loss.
We now have to find the local pressure loss and combine this loss with the global loss.
When a particular moves through the process gas, the gas does some work on the particular with as result a pressure loss, called drag. We can calculate this drag by assuming a wind field, calculate the flow path and integrate the drag along this path. By using different wind fields, which give different flow paths, it is possible to get an estimate of the total work done on the particular along the different paths. We assume that the particular is a sphere, so the drag on the sphere is known as function of the Reynold's number. The sphere is the best investigated body resistance in fluid. The Reynold's number is calculated along the flow path to evaluate if the assumption for the sphere's body resistance is a good approximation. If the sphere is very

small, the Reynold's number will be low, which tells us that the approximation is good. The different flow paths give different results for the total work for the particular, but only with a variation of plus minus 20 [%].

The calculation of the work done on the particulars.

The drag is calculated as the force acting on the immersed body, flowing in a gas stream. To transfer the results from experiments to the calculation, a dimensionless coefficient is used, called the drag coefficient C_d, which is a function of the shape of the body and the Reynold's number.

The equation for the drag coefficient C_d is:

$$C_d(\mathrm{Re}) = \frac{D}{\frac{1}{2}\rho V^2 A}$$

Where the D is the drag in [Newton], the ρ [kg/m^3] is the density of the fluid, the V [m/sec] is the velocity difference between the immersed body and the fluid and the A [m^2] is the frontal area exposed by the body to the flow direction. The C_d for a sphere can be found in many textbooks. Because the sphere is small, the Reynold's number becomes low, which means that the fact, that the particulars are not spherical and do not have a smooth surface, will not have a significant influence on the drag calculation, even when the flow is turbulent. We calculate the work done on the sphere along the flow path, choosing a wind field and integrating the drag along the path. $W(x) = \int_{s=0}^{s=x} D(s)ds$ where the $W(x)$ is the work along the way or flow path s [m]. The D is in [Newton] so the result is the work in [Joule]. If we place a control volume around a single particular, the particular will fall due to the gravity inside the control volume. Each time the particular falls the distance *"ds"* along the flow path, we have to do the work ΔW. The work over the distance *"ds"* is equal to $\Delta W=W(s+ds) - W(s)$ [Joule]. So each single particular creates a pressure loss: $\Delta p_{sphere} = \frac{\Delta W_{sphere}}{V_{volume}}$ The total work which has to be applied to move the sphere along the flow path, is then the sum of the work done on each of the particulars along their flight path. However, the sum of all the control volumes around each single sphere is equal to the larger control volume, which is the equipment wall. The total pressure loss is the additional pressure loss we measured between the equipment with and without product. It follows that the total pressure loss must be the sum of the work on each single sphere divided by the volume of the equipment. We now can find the number of particulars moving along the flow path at any given time.

$$p_{total} = \frac{\sum_{i=1}^{i=n} \Delta W_i \cdot i}{\sum_{i=1}^{i=n} V_{sphere}}$$

$$n = \frac{p_{total} \cdot V_{total}}{\int_{s=0}^{s=x} D(s)ds}$$

The equation for *"n"* is the general expression for how many particulars are flying around in the equipment at any given time. But we want to find the flux of particulars, the first derivative of the number in flight, which means we have to introduce the time. To do this we divided the flow in three parts. The first part is the coating columns, the second part is the up flight in the expansion chamber and the third part is the fall back to the down flow bed. We then have to find the work done in each part, calculate the volume for each part and find the resident time for the particulars in each part. We know that the total pressure is the sum of each of the three parts, which gives us the first equation. Where the n_1, n_2 and n_3 are the numbers of particulars in each of the three volumes, the τ_1, τ_2 and τ_3 are the average resident times for the particular in each of the three control volumes. We can now define a resident time between the three control volumes as $ratio_{1/2} = \tau_1/\tau_2$ and $ratio_{1/3} = \tau_1/\tau_3$, insert these in the equation and eliminate n_1. When we know n_1 we can calculate backward and determine n_2 and n_3 by using the resident ratio.

$$p_{total} = p_{part1} + p_{part2} + p_{part3}$$

$$p_{total} = \frac{\Delta W_1 \cdot n_1}{V_{volume1}} + \frac{\Delta W_2 \cdot n_2}{V_{volume2}} + \frac{\Delta W_3 \cdot n_3}{V_{volume3}}$$

$$n_1 \cdot \tau_1 = n_2 \cdot \tau_2 = n_3 \cdot \tau_3$$

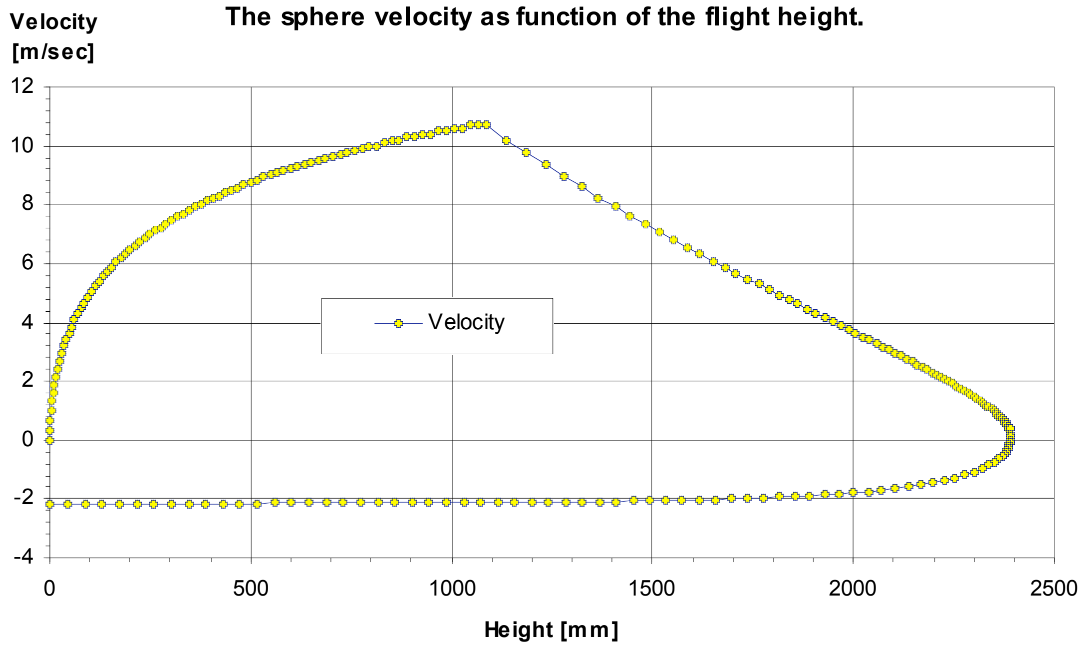
$$n_1 = \frac{p_{total}}{\frac{\Delta W_1}{V_{volume1}} + \frac{\Delta W_2}{V_{volume2}} \cdot ratio_{1/2} + \frac{\Delta W_3}{V_{volume3}} \cdot ratio_{1/3}}$$

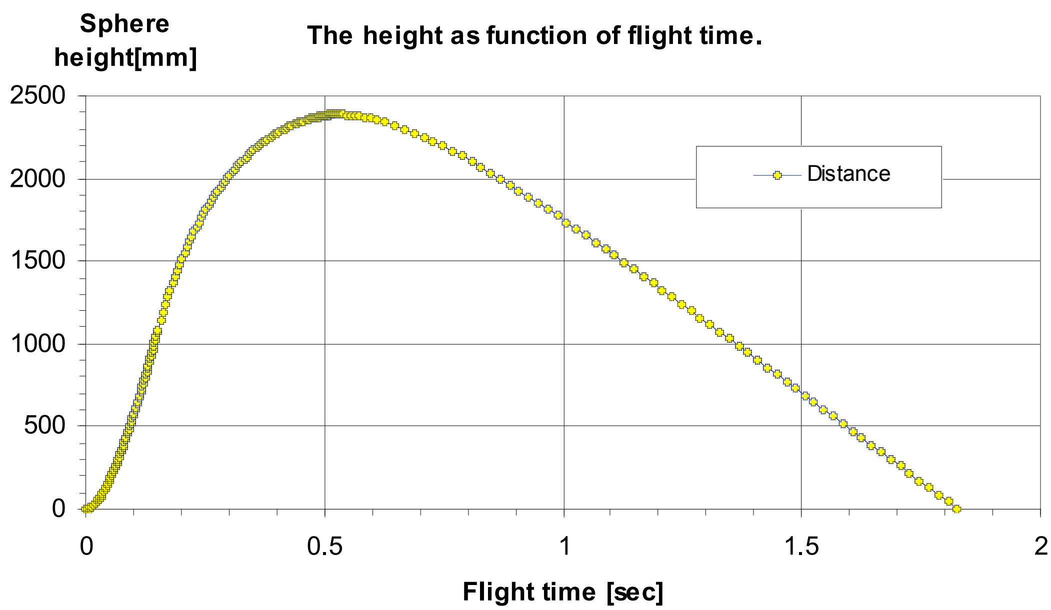
The result from a specific example.
The specific example is shown on the graph above. The batch weight was 300 [kg{nonpareil}] with an initial diameter of 550 [m]. The density of the pellets was1200 [kg/m³], the batch consisted of 2869714942 [nonpareil] which have a total surface area to be coated of 2727.2 [m²]. The coating process consists of four coating steps, active drug coat, subcoat, enteric coat and colour coat. The active drug coat has a solid content of 34.6 [%{solid}] which is not a normal coating solution. The Enteric and Colour coat have a solid content of 9.5 [%{solid}] for the Enteric and 10 [%{solid}] for the Colour coat, which is a normal solid content for most coating solutions. These results are close to what was found in experiments, which are mentioned in the beginning.

	Active coat	Sub coat	Enteric coat	Colour coat
Amount [kg]	300	644.5	863.3	1111.3
Solid sprayed [kg]	383.4	243.1	275.6	273.4
Solution sprayed [kg]	1107.9	1157.4	2909.8	1734.4
Time [sec]	19800	21600	61620	32400
Spray rate [g/min]	3357.3	3215	2833.3	3211.9
Coat growth rate [: m/h]	15.8	6.44	2.12	3.43

Calculation of the coverage rate.
As the volume flow pressure graph shows, most of the coating was done with a process air flow rate of 4000 [cfm] which is equal to 1.88 [m³{air}/sec] or 6768 [m³{air}/h], which is a high volume flow rate for the Precision Coater. The coating column diameter was120 [mm], so if all the process air passes through the columns, the velocity would be 16.9 [m/sec]. The empty bed pressure loss at 4000 [cfm] is measured to 12.4 [Inch of Water] which is equal to 3088 [Pascal]. The pressure losses with products are measured from 27.3 to 33.4 [Inch of Water] which gives a product transport pressure from 14.9 to 21 [Inch of Water]. This gives us the total pressure for product from 3711 [Pascal] to 5231 [Pascal]. This large difference in transport pressure happens, when the particulars are covered with the first layer of coat; they become more free flowing, so more product will flow from the down flow bed into the coating column. But because the system is made in such a way that the volume flow is kept constant, the air has to transport more product, and therefore the pressure loss increases. The rate of product transported is linear (or close to linear) with the transport pressure, so the ratio in transport is from a product flux equal one to a flux which is 1.4 or 40 [%{product flow}] higher. Using the flow model for a single sphere with different start conditions, the average flight path of the particulars can be calculated. We can now make an estimate of the amount of particulars in the coating column, in up flight and down flight. The maximal surface flux through the equipment is 70.96 [m²{surface area}/sec] and the minimal flux is calculated to be 50.35 [m²{surface area}/sec]. If we assume that the polymer’s thickness for all four coating steps is 330 [n m] and the coat is applied as a monolayer, which is the worst-case scenario, we obtain:

[%{single layer}]	Active coat	Sub coat	Enteric coat	Colour coat
Maximal flux	66.1	38.4	15.3	28.8
Minimum flux	93.2	54.2	21.5	40.6





The first graph displays the average velocity of the pellet as function of the height from origin and the second graph displays the pellet's position from origin as function of the elapsed time from start.

Only the active drug coat which has a solid content of 34.6 [%{solid}] comes near full coverage. But if we assume that there are four to six polymer layers per coat application, we arrive at a maximal of 20 to 25 [%{coverage}]. When we did the same kind of experiment in Bubendorf, Switzerland, we came to a result of 4 to 8 [%{coverage}]. We used then a 400 [□ m] pellet and not, as here, a 550 [□ m] pellet. The surface area of a 400 [□ m] pellet with the same pellet flux as the 550 [□ m] pellet is 2.6 times larger. The analysis and calculation in this investigation show nearly the same results as in the investigation from ten years ago, which is mentioned in the beginning of this publication.

We obtained other interesting results from this analysis. The calculation predicts that out of the 2869714942 pellets in the batch, the number of pellets which are in free flight at any given time can be broken down in the three parts as:

	Coating column	Upward flight	Downward flight
Prediction maximal	89030329	37075644	10330236
Prediction minimum	63169138	26306052	7329548

Calculating the percentage of the total batch in free flight, we get that there are between 2.3 to 3.1 [%{free flight/total}] in the coating columns and from 1.17 to 1.65 [%{free flight/total}] in upward or down ward flight at any given time. This can also be calculated as how much of the volume in the three parts is occupied with solids (pellets) compared with the total volume or how the image of the pellets in free flight would appear when we look into the equipment. This calculation shows, that the average concentrations inside the coating columns are between 4.8 and 6.3 [%{solid volume/total volume}]. In the expansion chamber the particulars, which are in the up and down flight, occupy from 0.021 to 0.03 [%{solid volume/total volume}]. This is indeed a small number, the concentration of 0.021 [%{solid volume/total volume}] which means that only a 1/ 5000th part of the total volume is occupied by a sphere (pellets). Inside the coating column the concentration varies from near 60 [%{solid volume/total volume}] in the vicinity of the spray nozzle to something below the average 4.8 and 6.3 [%{solid volume/total volume}] at the outlet of the coating column. If we assume that the concentration is not strengthened by the low pressure zone created by the process air combined with the low pressure from the atomizing air, but only diluted or stretched by the longitudinal acceleration, we find the concentration 30 [%] at 10 [mm] from the origin, 20 [%] at 20 [mm] and 10 [%{concentration}] at160 [mm] from the origin, the insert corner.
We also get information about the resident time in the different zones. The resident time in the spray zone is around 0.05 [sec], the flight time through the coating column is 0.15 [sec], the upward flight time is 0.37 [sec] and the time from apogee to the product layer in the down flow bed is around 1.3 [sec]. The total flight time from the corner of the insert until the pellet is back in the down flow bed takes 1.827 [sec] in average.

Conclusion.
The analysis shows that the flight path calculation, combined with the measurement of the additional pressure loss, can be used to evaluate the coat coverage. The measured pressure loss over the coater is the sum of the equipment pressure loss and the product pneumatic transport pressure loss. The product flow through the equipment can be evaluated in real time by calculating and displaying the product pneumatic transport pressure loss in real time during the coating process.
The analysis also shows, that only a small part of the pellet’s surface is covered by each passing through the coating zone.
To avoid attrition, the coating has to be efficient from the start of the coating process; there is no risk for a too high coat growth rate.