# Wurster coating PAT monitoring by NIR: calibration vs. PCA trend approach

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## PURPOSE

To follow the wurster coating process trend using NIR technology and to determine real-time the deviations from the correct process path. To establish the correlation between the sprayed mixture quantity and the response of the NIR device. To demonstrate the possibility to obtain good process monitoring and a correct process trend determination with different coating materials. To find a mathematical and statistical approach to the in-process control using derivative method, PCA calculation and trend approach with the aim of a full integration of the PAT control into the fluid bed software.

## METHODS

Inert sugar spheres 20-25 mesh was used in this study as coating cores. The set of processes were performed in a Freund-Vector VFC Lab 3 fluid bed, equipped with Wurster partition, Freund-Vector Accelerator 1 diverter system and Viavi MicroNIR PAT U device. The NIR device is connected to the fluid bed with a welded flange.
The load of dry cores was adjusted for the type of equipment to achieve appropriate product movement and the correct quantity was determined to be 3500 g. Three repetitions of the same process were made for every coating agent to demonstrate the robustness of the method. Data were analyzed and processed using Unscrambler© software.

*Table 1 - Coating Suspensions*

| *Component* | *Quantity (g)* | *%* |
|---|---|---|
| Opadry II blue | 225 | 15 |
| Opadry II green | 225 | 15 |
| Opadry II clear | 225 | 15 |

## RESULTS

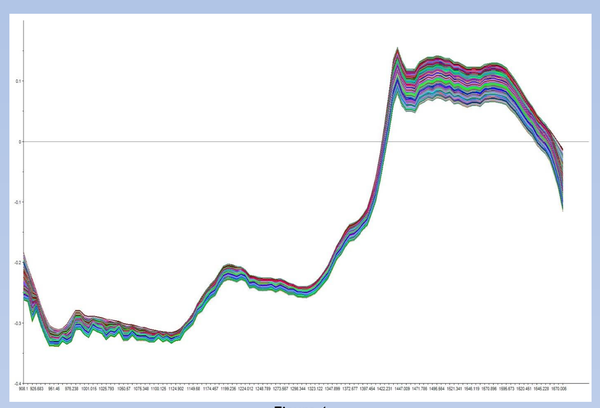

Figure 1

*Table 2 - Process Parameters*

| | | | |
|---|---|---|---|
| Inlet air Temp. (°C) | 55 | Process time (min) | 130 |
| Product Temp. (°C) | 42 | Initial LOD (%) | Approx 1.5 |
| Airflow ($m^3$/hr) | 120-130 | Final LOD (%) | Approx. 1.5 |

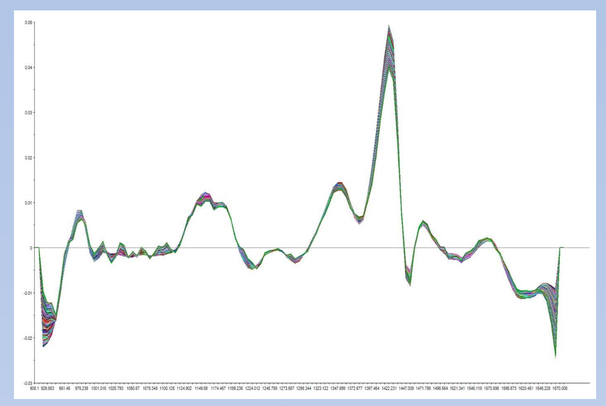

Figure 2

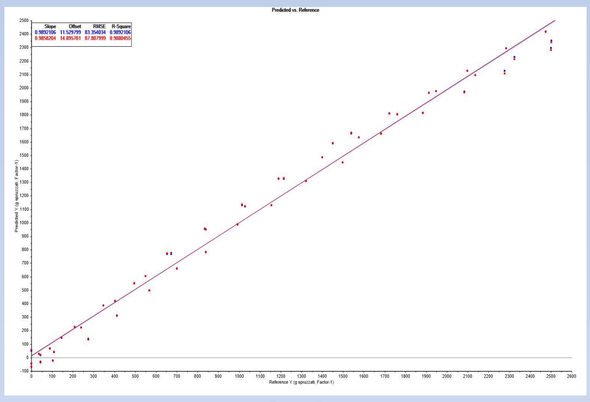


Figure 3

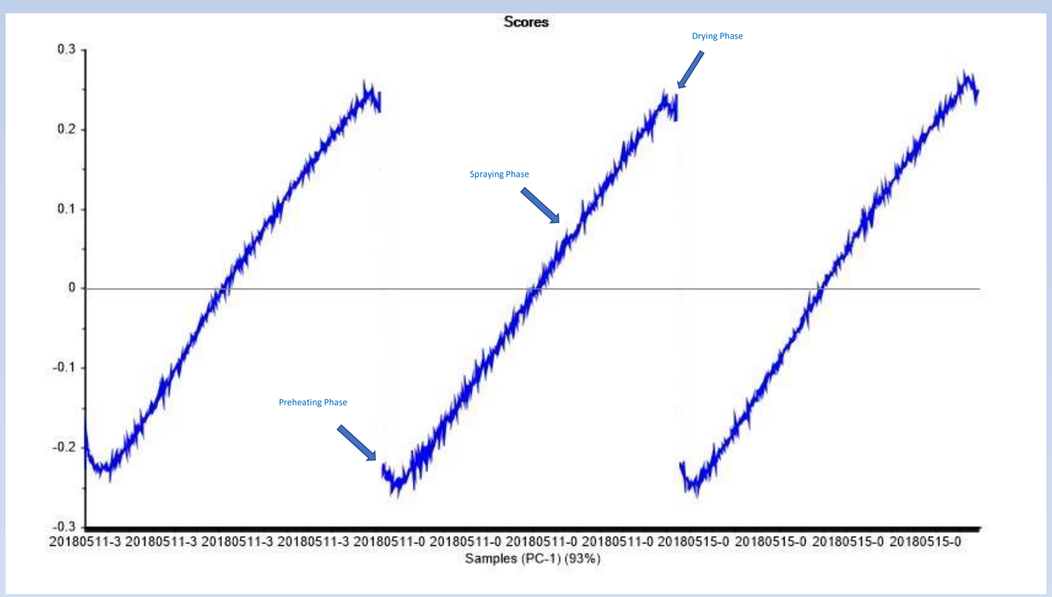


Figure 6

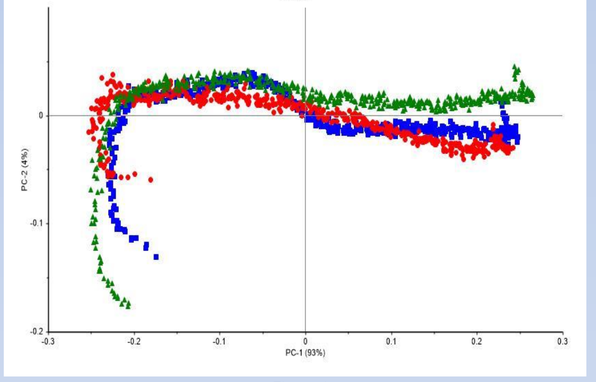


Figure 4

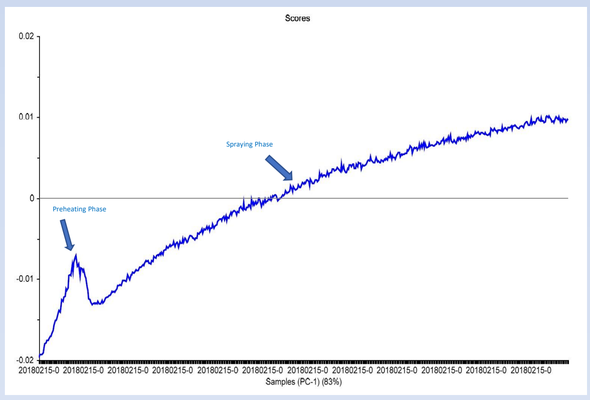


Figure 5

Figure 7

The data presented refers to the Opadry II blue coating agent. The Absorbance curve and the 1st derivative, corrected by SNV algorithm are shown in Figures 1-2. The correlation between the real amount of srpayed coating suspension and the predicted value is shown in fig. 3. The high $R^2$ shows that there is a strong correlation between the real data and the response of the NIR, confirming that the model is robust. The corrispondence between the sprayed amount and the NIR response is a strong indicator of the efficiency of the process and of the real quantity of coating material that effectively covers the cores.
In fig. 4 the PCA plot of three different batches are shown overlapped to demonstrate that the points are sequentially aligned and the different batches follow a highly comparable path. The PCA scores of a single batch is shown in fig. 5, it is possible to see a very clear trend and the preheating phase is discernable from the spray phase. In fig. 6 is shown a comparison between the scores of the PCAs of the three batches, it is possible to notice that the trend of the three processes is highly comparable. In fig. 7 is shown the installation configuration of the NIR probe on the fliud bed.


Freund-Vector VFC LAB 3

Viavi MicroNIR PAT-U Equipment

## CONCLUSIONS

The process shows a very strong correlation between the absorbances read by the NIR instrument and the quantity of coating suspension sprayed. It is clearly visible the similar trend of the process repetitions. This indicates that the model is reproducible. It is possible to distinguish the different phases of the coating process, preheating, spraying, drying. The reproducibility of the process in terms of trend can allow a process control without the need of a constant calibration. It is possible to assume that every significant deviation from the trend can be considered a drift of the process and immediately indicates that a correction is necessary to achieve a good quality product. A future developement of this study can lead to a real time control of the process in terms of product carachteristics instead of the process parameters, with the aim of an integration with the automatic control system.