Monitoring Fluid Bed Coating Processes with Real Time Imaging

Authors: P. Cruise, Ian Jones, C Cortazzo, Joachim Fröhlich, Lilia Sprich, Raoul Pila

Introduction

Iinnopharma Labs Eyecon™ inline particle characterisation technology enables customers to measure particle size and shape during fluidised bed granulation & coating, spheronisation and continuous fluid bed drying processes. This study examines the effectiveness of a particle characterising technology to capture particle images and to calculate particle growth during a fluidised bed spray coating process.

Experimental Plan

The objective of this study was to examine if the Eyecon has the ability to track the increase in particle growth over time as a result of the spray coating process.

Materials & Equipment

The experiment was carried out using a pilot scale fluidised bed coater (Glatt, model GPCG15 with Wurster insert). The Wurster coater used was configured as follows:

- Wurster with bottom plate C/G
- Wurster pipe 35mm
- HS 04 spray nozzle with 4.0mm liquid insert
- Bonnet 250μm

Metoprolol Pellets 77.7% with a starting diameter 516.4μm were used as the substrate material. The spray solution comprised of Eudragit FS 30D / Talkun / Triethyl Citrate and purified water. 8.75kg of spheroids were mixed with 20% concentration of coating material. The total mass of coating fluid added was 18.375 kg. The addition rate was 100-120g/min and the total spray time of the coating process was 153 minutes while drying and cooling time was 6 minutes.
Measurement of spheroids was carried out using the Eyecon Particle Characteriser. The Eyecon was developed by Innopharma Labs as PAT (Process Analytical Technology) for the pharmaceutical industry. Its non-product-contact design allows it to be easily integrated into manufacturing equipment either in-process or at material outlets for real-time monitoring and control of particle size. The Eyecon was integrated onto a standard viewing port and acquired images of spheroids through the port.

Results & Discussions

Samples from the start and end of the process were selected and analysed. Table 1 shows the results of Eyecon and sieve analysis measurement data of the sample before and after the coating process. This measurement data allows for the calculation of particle growth as a result of spray coating. A noticeable increase in particle size distributions before and after the spray coating process, as recorded by the Eyecon, can be seen in figure 1.

Table 1: particle size growth as recorded by Eyecon inline and by sieve analysis.

<table>
<thead>
<tr>
<th></th>
<th>Before Coating (μm)</th>
<th>After Coating (μm)</th>
<th>Particle Growth (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sieve Analysis</td>
<td>516</td>
<td>598</td>
<td>82</td>
</tr>
<tr>
<td>Eyecon™</td>
<td>472</td>
<td>557</td>
<td>85</td>
</tr>
</tbody>
</table>

Figure 1: PSD distribution of the sample at the beginning and end of the fluidised bed spray coating process
Figure 2 highlights the ability of the Eyecon to monitor the growth of spheroids over time throughout the spray coating process. This real-time information could provide important data for the optimisation of the process.

![Increasing Particle Growth during the Spray Coating Process](image)

*Figure 2: Particle size growth during the spray coating process using real-time measurement data from the Eyecon Particle Characteriser.*
Conclusion

The results of this study show the Eyecon’s ability to monitor particle size growth during a fluidised bed spray coating process. The ability to monitor particle growth at discreet time periods will allow for more in-depth understanding of the process and will aid in the identification of process perturbations and enhancing process robustness.

As a means of start and end point measurement the Eyecon provides accurate results equivalent to sieve analysis but has the added benefit of in process particle growth monitoring.

The Eyecon particle characteriser successfully captured images and subsequently calculated particle size distributions for the sample materials in a rapid and accurate manner. It can be concluded that the Eyecon can successfully track the increase in particle growth over time as a result of the spray coating process.

For More Information on Eyecon™ Please Contact

Mike Mulcahy, Sales Director
Innopharma Labs
mulcahym@innopharmalabs.com
+353 1 485 3346