Abstract

The using of near infrared process analytical technologies (PAT) in pharmaceutical manufacturing drying processes

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Introduction

Drying processes are very widely-used operating units in pharmaceutical manufacturing processes. New innovative Process Analytical Technologies (PAT) systems have been introduced, specifically targeted for the pharmaceutical industry. Pfizer have taken leadership roles to advance the technology by coordinating with instrumentation companies for newly-designed instrumental solutions. This presentation will present some examples of using near infrared (NIR) spectroscopy for monitoring on-line drying in current pharmaceutical manufacturing processes, including fluid bed drying, tray dryers and tumble dryers.

Materials and methods

Operation of three pharmaceutical drying processes, including drying an active pharmaceutical ingredient (API) in a tumble dryer, a wet granulation drying process in a fluid bed dryer and drying a drug product in a tray dryer was used for NIR PAT method development. An EXPO NIR Analyser (a photodiode array type NIR spectrometer) with probe, operating in the wavelength region from 1000 nm-1750 nm, and multivariate analysis algorithms were used for PAT applications in the fluid bed dryer and tumble dryer aspects of pharmaceutical manufacturing processes. An ABB FT-NIR spectrometer with gas cell was used for PAT applications in the tray dryer aspect of the pharmaceutical manufacturing process.

Results and discussion

All the NIR spectra were pre-processed with 2nd derivative data pretreatment before analysis. Partial least squares (PLS) algorithm was used to develop the NIR calibration models for on-line

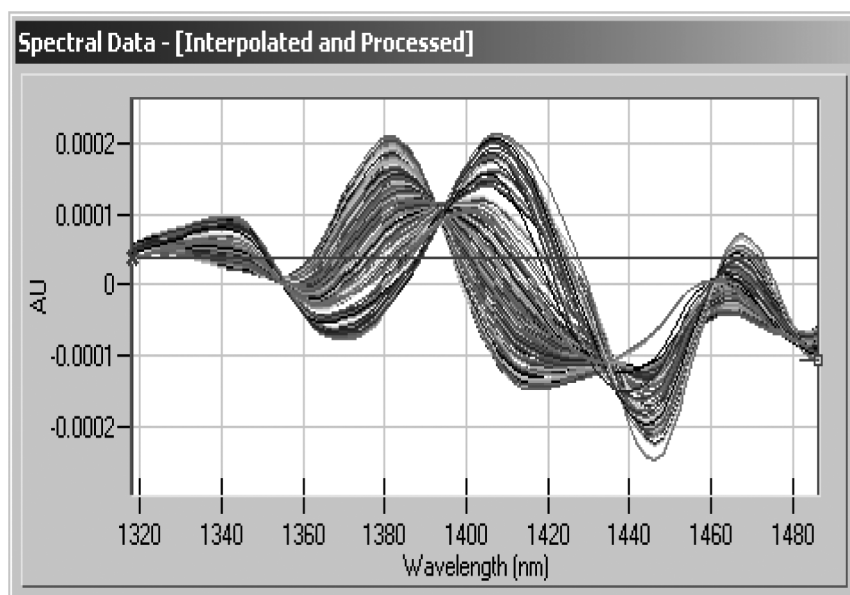**Figure 1.** NIR Probe attached to fluid bed dryer.

Figure 2. NIR 2nd derivative spectra of moisture peak.
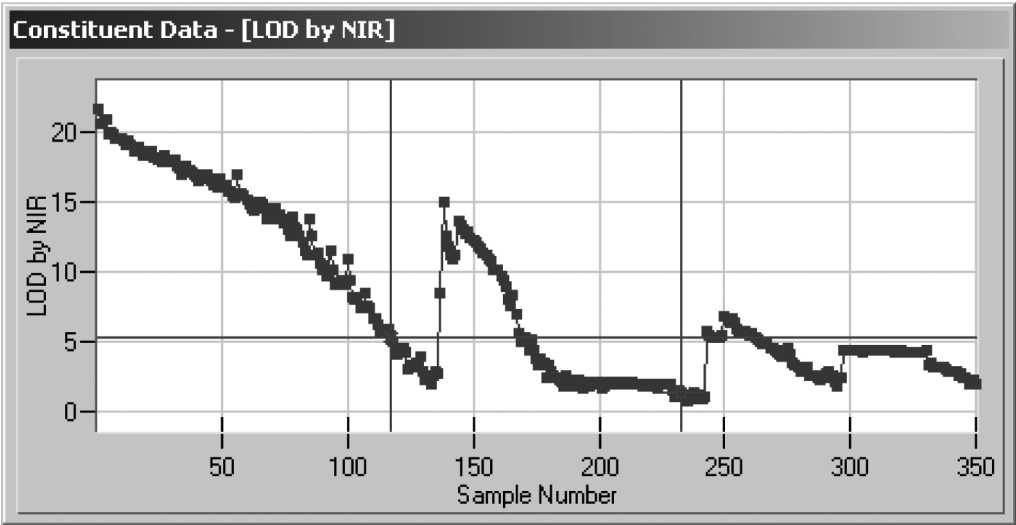
Constituent Data - [LOD by NIR]

Figure 3. NIR drying profile of wet granulation drying in FBD.

real-time prediction of process end-point. Preliminary results are shown in Figures 1-3, and Table 1.

These include the NIR installation (Figure 1), NIR spectra meaured in FBD (Figure 2), NIR method performance compared with LOD (loss on drying) method (Table 1) and NIR drying profile of wet granulation drying in the fluid bed drier (FBD, Figure 3).

Table 1. NIR method performance (drying endpoint prediction).

Bowl	Lot #	Mix#	LOD (COMP)	NIR	Residual
A	27414i	3	1.316	1.673	0.357
	27424i	3	1.227	1.494	0.267
	45164i	1	1.024	1.052	0.028
B	27634i	1	1.757	1.794	0.037
	27664i	3	1.882	1.869	0.013
	27684i	2	1.805	1.677	0.128