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Lubricant-Sensitivity Assessment of SPRESS® B820 by Near Infrared Spectroscopy: A Comparison of Multivariate Methods

Abstract

The aim of the present investigation was to compare the predictive performance of PCR and PLS calibration models based on near-infrared spectroscopy (NIRS) data used to monitor lubrication conditions impact on the tablet breaking force (TBF) of SPRESS® B820.

Disciplines

Pharmacy and Pharmaceutical Sciences

Comments

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Purpose:

The aim of the present investigation was to compare the predictive performance of PCR and PLS calibration models based on near-infrared spectroscopy (NIRS) data used to monitor lubrication conditions impact on the tablet breaking force (TBF) of SPRESS® B820.

Methods

2³ Full Factorial Design

- Model material: SPRESS® B820 (pregelatinized starch)
- Design variables
 - Magnesium stearate fraction
 - Blending time
 - Compression force
- Response variable
 - Tablet breaking force

Tableting

- 10-station instrumented rotary tablet press
- Standard concave, 10mm, round, B tooling
- Target tablet weight: 500 mg

NIR Analysis

- Mode: reflectance
- Spectral range: 1117.00 nm – 2499.50 nm

Tablet Evaluation

- Automatic breaking force tester

Spectral Data Processing

- Baseline correction
- Savitzky-Golay method coupled with 2nd polynomial order
- Multivariate Calibration Models
 - Qualitative: Principle component analysis (PCA)
 - Quantitative: Principle component regression (PCR) Partial least squares (PLS)

- Prediction Performance Testing
- Independent validation data set

Results

Figure 1. Typical NIR spectrum of magnesium stearate (MgSt).

Figure 2: Processed NIR spectra of SPRESS® B820 tablets prepared at 15 kN, 20 kN, and 25 kN from the powders lubricated with 0.25% w/w (blue lines), 0.5% w/w (pink lines), and 1% w/w (green line) MgSt for 2, 5, or 10 min. . Arrows indicate peaks with varying NIR spectra as a function of the added MgSt fraction, blending time, and compression force.

Model name – MgSt fraction	Number of PCs used	Calibration		Validation	
		R ² Value	RMSEC	R ² Value	RMSEV
PCR-All	7	0.7853	14.66	0.7788	14.92
PCR-0.25%	5	0.9410	6.52	0.9383	6.73
PCR-0.50%	6	0.9700	3.61	0.9675	3.80
PCR-1.00%	7	0.8839	9.78	0.8778	10.11
PLS-All	7	0.8525	12.15	0.8404	12.67
PLS-0.25%	3	0.9392	6.63	0.9361	6.84
PLS-0.50%	5	0.9707	3.57	0.9679	3.77
PLS-1.00%	5	0.9176	8.25	0.9052	8.91

Table 1: Near infrared spectroscopy (NIRS) based modeling details developed using partial least square regressions (PLS)-1 and principal component regression (PCR).

Figure 3: Correlation of measured and predicted values of TBF of SPRESS® B820 from the independent dataset of tablets prepared under various conditions, using different PCR calibration models. SPRESS® B820 was lubricated with 0.25, 0.5, or 1% w/w MgSt for 2 min, 5 min, or 10 min. The powders were compressed at 15 kN, 20kN, and 25kN. TBF predicted from (A) PCR-1 calibration model calculated from samples lubricated with 0.25, 0.5, and 1% w/w MgSt; (B) PCR-1 calibration model calculated from samples lubricated with 0.25% MgSt; (C) PCR-1 calibration model calculated from samples lubricated with 0.50% MgSt; (D) PCR-1 calibration model calculated using sample lubricated with 1.00% MgSt.

Figure 4: Correlation of measured and predicted values of TBF of SPRESS® B820 from the independent dataset of tablets prepared under different experimental conditions using partial least square regression (PLS-1) calibration models. SPRESS® B820 was lubricated with 0.25, 0.5, and 1% w/w MgSt for 2 min, 5 min, and 10 min. The powders were compressed at 15 kN, 20 kN, and 25 kN. TBF predicted from (A) PLS-1 calibration model calculated from samples lubricated with 0.25, 0.5, and 1% w/w MgSt; (B) PLS-1 calibration model calculated from samples lubricated with 0.25% MgSt; (C) PLS-1 calibration model calculated from samples lubricated with 0.50% MgSt; (D) PLS-1 calibration model calculated from sample lubricated with 1.00% MgSt.

Conclusions

The current study emphasizes a careful selection of multivariate calibration methods as well as lubrication fraction range during the calibration and validation stages, as it may have a significant impact on the prediction performance of the developed models.