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Detection of Moisture Using a NIR Filter Photometer on Dynamically Moving Particles in a Rotor Processor

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PURPOSE

Following the FDA's PAT (Process-Analytical-Technology) initiative in the early 2000s, numerous studies have been conducted utilizing full spectrum near infrared (NIR) sensing technology to monitor moisture in granulation and drying processes. In order to achieve good "mapping" of the process, the sample being presented to the sensor needed to be "at rest" and in contact with the sensor/optics in order to achieve usable spectra. This study investigated the feasibility of using a NIR filter photometer on a dynamically moving product bed not in contact with the sensor.

METHOD

Separate granulations of lactose (Lactose 312, Foremost™), corn starch (Pure-Dent® Starch B815, Grain Processing Corporation), and microcrystalline cellulose (MCC, Emcocel® 50M, JRS Pharma) were granulated with 5% polyvinylpyrrolidone (PVP, Plasdone™ K-29/32, Ashland™) aqueous solution in a conical rotor (GXR-35 insert in VFC-Lab 3 Flo-Coater®, Freund-Vector; Figure 1). Formulations of 70% lactose/30% MCC and 85% lactose/15% starch were also granulated with 5% PVP. Absorbance values of the moving product bed were recorded using a NIR fiber optic photometer (RS1000, Innovative Technologies Group; Figure 2) located approximately four inches above the product surface. Granulation samples were taken throughout the process and LOD (Loss-On-Drying) measurements were made with a moisture analyzer (Mark 3 LTE, Sartorius). Absorbance values and LOD measurements were correlated to make corresponding calibration curves.

Figure 1

Figure 2

RESULTS

Figure 3

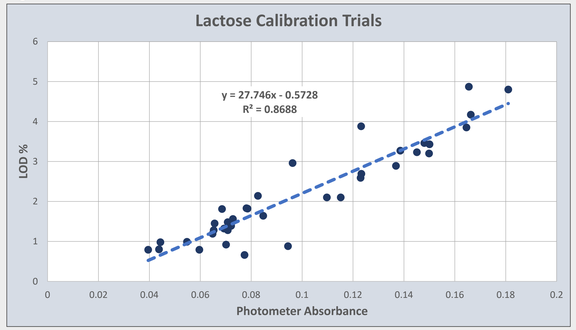


Figure 4

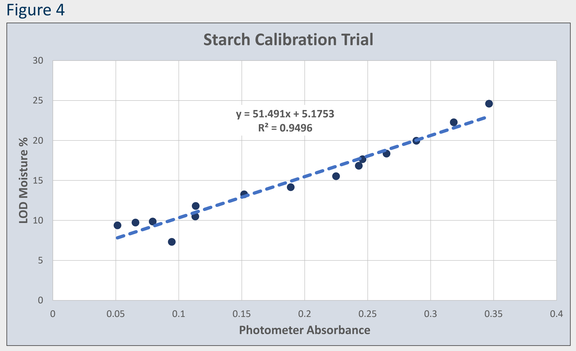
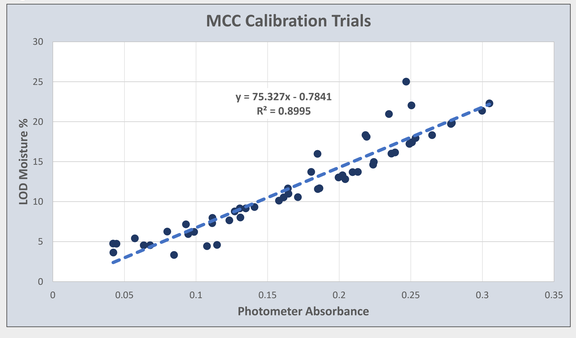


Figure 5



RESULTS

Figure 6

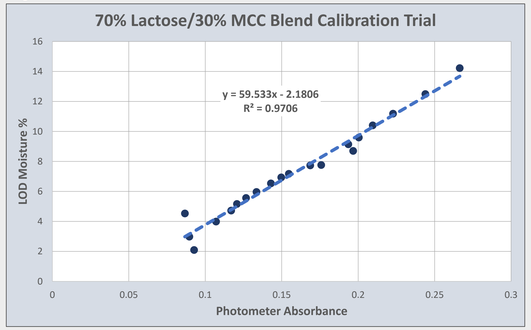


Figure 7

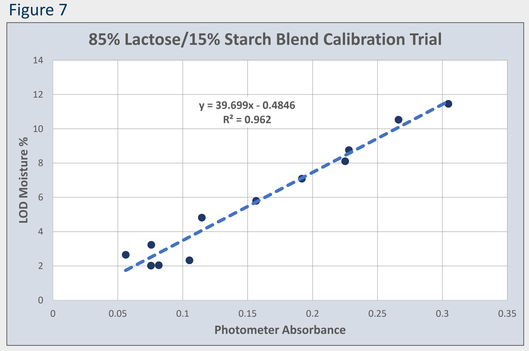


Figure 8

Lactose Validation Trial			
Time Stamp	LOD %	Moisture %	Error
1:44	0.96%	1.35%	0.39%
1:50	1.33%	1.14%	0.19%
1:56	1.86%	1.83%	0.03%
2:02	2.47%	2.25%	0.22%
2:08	2.81%	2.80%	0.01%
2:14	3.39%	3.02%	0.37%
2:21	3.41%	3.09%	0.32%
2:28	3.96%	3.60%	0.36%
2:34	2.88%	3.17%	0.29%
2:40	2.45%	2.87%	0.42%
2:46	3.14%	3.45%	0.31%
2:53	5.01%	4.40%	0.61%
3:00	6.84%	5.78%	1.06%
3:07	4.07%	4.33%	0.26%
3:14	1.39%	1.43%	0.04%

Error = *abs(photometer % - LOD %)*

RESULTS

Figures 3-5 display the data gathered from the trials using pure materials. Best fit lines for each material resulted in different slope values for each material. Programming the calibration equation into the photometer allowed it to display moisture values instead of absorbances.

Figures 6 and 7 display the data gathered from trials using combinations of the pure materials. These trials tested the theory that the equation for a blend of material can be predicted if the equations for the pure materials are already known. This proved false. The slope of an equation for a blend of materials will skew toward the slope of the pure material which has the greatest capacity for absorbing moisture.

Figure 8 displays the results of a validation trial conducted with lactose to test the equation that was programmed into the photometer. Comparison of the photometer moisture display to the LOD moisture shows a nominal 1% error or less.

CONCLUSIONS

- Moisture content of dynamically moving materials in a spherical granulation process can be successfully monitored using a single wavelength filter photometer.
- Creating a moisture model is very simple using a filter photometer as compared to a full spectrum NIR instrument.
- Any change in formulation will likely require a new moisture model.

ACKNOWLEDGEMENTS

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