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Short communication

Real-time NIR monitoring of fluid-bed coating process of glucomannan

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Introduction

Obesity, recognized as a chronic disease by the WHO in 1997 and later classified by the EU in 2021 as a "chronic relapsing disease," underscores the need for effective preventive strategies, particularly dietary regulation and physical activity (Burki, 2021).

European Food Safety Authority (EFSA) in 2010 endorsed claims on Konjac glucomannan (GLM) in weight reduction and maintenance of normal glucose, triglyceride and cholesterol levels (Kuczora, 2015). Strong water-absorbing properties of GLM are beneficial for its functional applications, but a significant drawback due to the risk of choking when ingested as powder (Mortensen et al., 2017). Several approaches, including chemical modifications and physical treatments, have been investigated to enhance safety and functionality of GLM (Tatirat et al., 2012).

A promising strategy to overcome the limitations of GLM involves fluid bed coating with an ethanolic solution of Eudragit EPO (EPO), producing a protective water-resistant coating. The Wurster technique, yields a GLM powder with enhanced safety and preserved functional properties. Notably, the coated particles demonstrated favorable flow characteristics, comparable to those of unmodified GLM, suggesting that this approach effectively balances usability and risk mitigation (Gigovski et al., 2023).

Considering the complexity of the Wurster fluidization coating process, the present study aimed to evaluate near-infrared (NIR) spectroscopy as a process analytical technology (PAT) for real-time monitoring and reliable end-point determination, thereby supporting more consistent GLM formulations in terms of film quality.

Materials and methods

Konjac mannan, glucomannan (GLM, mol. weight 80.000-120.000 Da) was obtained from Straqim S.A., Switzerland. Ethanol (96% v/v) was purchased from Alkaloid AD Skopje, N. Macedonia and Eudragit EPO (EPO) was provided from Evonik Industries (Darmstadt, Germany). All other chemicals used were of pharmaceutical grade and were used without further modifications.

The fluid-bed coating of raw GLM was conducted using a VFC Lab Micro Flo Coater (Freund-Vector Corporation, USA). For the Wurster

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coating process, a 4% EPO was applied (sample G-EPO4). The Wurster granulation was performed with: air flow 17 LPM; inlet temperature 45 °C; nozzle air 75 mBar; pump speed 10 rpm; granulation time 10 min/cycle. NIR probe 1700ES (Viavi Instruments, USA) was flush-mounted on the side viewing port of the fluid bed container. NIR spectra were continuously acquired at each 20 s of the process (acquisition time 10 s, 900-1260 nm spectral range). The collected spectra were preprocessed using standard normal variate (SNV) transformation and first derivative techniques. Subsequently, principal component analysis (PCA) was applied to the processed data using Simca 14.1 software (Umetrics, Sweden).

Granule morphology was examined before, during, and after coating using a Zeiss Stemi 305 optical microscope (Germany) at 2.5 and 5 magnifications, with image analysis performed in Zeiss Zen 3.8 software. FTIR and Raman spectra of uncoated and coated samples were obtained using Cary 630 (Agilent, Germany) and ATR 3500 Optosky (China), respectively.

Results and discussion

The morphology of GLM granules coated with EPO (sample G-EPO4) revealed irregular, translucent particles with predominantly angular to subrounded shapes. Pronounced agglomeration and surface roughness were evident, suggesting particle adhesion on the GLM surface. The coated granules formed clusters with a broad size distribution from 500 µm to over 1000 µm across their largest dimensions, indicating substantial variability in agglomeration.

FTIR and Raman spectra confirmed successful EPO coating of the particle surface, with no detectable characteristic GLM bands, indicating complete surface coverage by the polymer.

The PCA model was constructed from 984 spectra continuously acquired during online monitoring. Outliers beyond the 99% confidence limit were excluded using the T^2 Hotelling approach. The final model, based on eight principal components, explained 99.5% of the total variability, with PC1 and PC2 accounting for 81%. Only PC1 was directly associated with process progression. The loading plot indicated that spectral regions at 1150 nm and 1350 nm (originating from EPO) correlated positively with coating progression, while the 1250 nm band (originating from GLM) was inversely related and gradually disappeared as the coating process progressed. Analysis of PC1 scores suggested that the coating process reached its end-point at approximately 210 minutes, after which no further spectral changes were observed, assuming complete GLM coverage.

Conclusion

The study demonstrated favorable morphological characteristics of coated GLM and confirmed EPO deposition via FTIR and Raman analysis. The PCA model demonstrated that NIR spectroscopy can serve as an efficient PAT for real-time, on-line monitoring of the Wurster fluidization coating of GLM with EPO. By capturing chemical signals from the granule surface, NIR enabled reliable end-point determination, supporting improved process control and reproducibility.

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