ANALYSIS OF TABLET FILM COATS USING LIBS

APPLICATION NOTE LIBS-020

The majority of pharmaceutical tablets on the market are film-coated, which serves several purposes, including taste masking, dissolution modification, and the protection of active pharmaceutical ingredients (APIs) against air, moisture and light. The photo-stability of film-coated tablets often depends on the thickness of the coating pigment. LIBS analysis was applied to tablets in this experiment to demonstrate effective monitoring of coating composition, thickness and uniformity with minimum sample preparation.

Samples

Core tablets with and without a film coat were obtained for LIBS analysis. The core tablets contained a photosensitive API, microcrystalline cellulose, sodium carboxymethylcellulose, anhydrous lactose, and magnesium stearate. The film coat was composed of Opadry® HP aqueous film coating, which contains both titanium (Ti) and iron (Fe), and pigments containing 2, 4 or 7% Fe_2O_3 (w/w). The film coats were applied to the core tablets at w/w levels of 2, 3 or 4%.

Analysis Method

A LIBS instrument with the following settings was used to analyze the core tablets and film-coated tablets:

Laser	Spectrometer	Sampling	Data
Energy = 100mJ	Delay = 1 µs	33 sites	Fe (405 nm), Ti (391 nm)
Rep rate = 2 Hz	Gate = 3 µs	25 shots/site	Net signal, peak height

Solid tablets were analyzed directly with LIBS with no sample preparation (i.e. no digestion or no surface preparation).

Results

In Figure 1, LIBS spectra from core and film-coated tablets are compared. The LIBS optical emission spectra collected showed a distinct difference between the tablet core and the film coating. The coating showed Ti and Fe, as expected from the Opadry HP, while in contrast, the core did not show these elements – and provided a low background where the Ti and Fe signals were chosen.

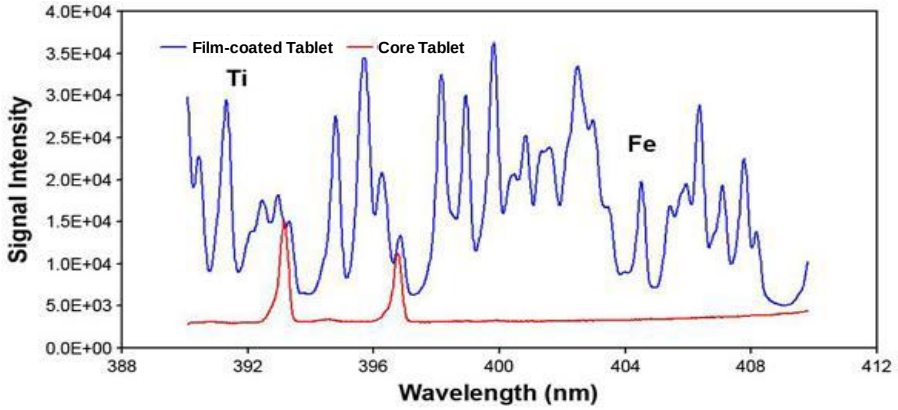

Figure 1. LIBS Analysis of Tablet Film Coating and Core Tablet

In Figure 2, the profile of iron (Fe) signal as a function of laser pulse number shows that the number of laser shots required for a coating to be penetrated is directly proportional to the coating concentration applied, and hence coating thickness. With 4% wt/wt coating, it can be seen that the first 3 laser shots did not penetrate through the film coating – as the Fe remained constant. As subsequent shots were taken, less Fe from the film coating was observed, indicating penetration into the core of the tablet, until eventually only the core tablet was being analyzed when the Fe level stabilized. It was found that the area under the curve was directly proportional to the amount of coating.

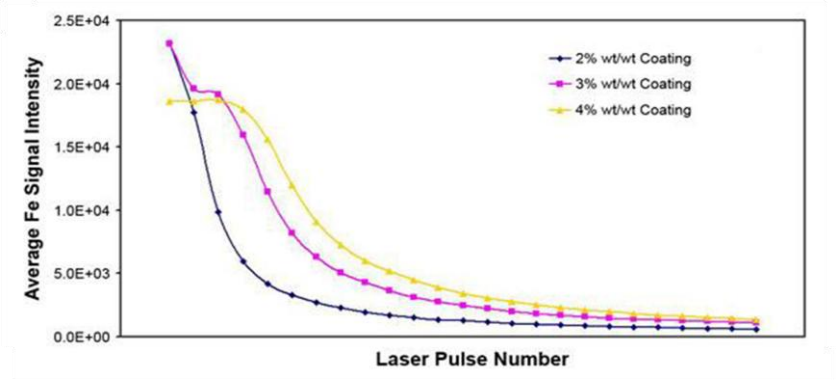

Figure 2. LIBS Analysis of Film-Coat Thickness

The film coat thickness at different locations on the tablet was also investigated to determine if the coating thickness varied spatially on the tablet. In Figure 3, the data showed there to be a statistically significant difference in coating distribution for 4% wt/wt, with the tablet edges having a thicker coat than the center of tablet. Spatial variability was not statistically significant at 2% wt/wt coating suggesting this may be a better concentration to apply for uniformity in coating – or a different coating procedure may need to be applied to the core tablet if high wt% coatings are required.

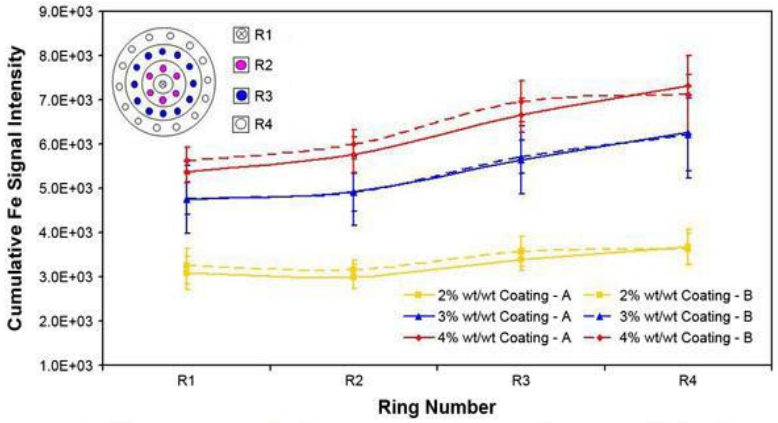

Figure 3. LIBS Analysis of Film-Coat Distribution

Conclusions

LIBS analysis of pharmaceutical tablets was effective at monitoring film-coating thickness by assessing the laser pulse number required to penetrate to the core tablet. The distribution of film-coat was also readily determined by spatially resolving the cumulative iron (Fe) signal on a tablet. In these particular experiments, LIBS analysis found that the film-coat thickness on the core tablets was proportional to the w/w level at which it was applied, with more coating applied to the edges than the center of the tablet. These LIBS results were obtained with no tablet preparation/processing required, demonstrating the rapid diagnostic capability of the method for tablet coating research and quality assurance/control.

Reference

Madamba, M., Mullett, M. W., Debnath, S., Kwong, E. Characterization of Tablet Coatings Using Laser-Induced Breakdown Spectroscopy, *AAPS PharmSciTech,* 2007, 8, E1-E7.

