DVS Application Note 116

Dynamic Vapor Sorption as a PAT Tool to Assess Tablet Coating

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Film coating in pharmaceuticals is a functional, performance-defining parameter in tablet manufacturing particularly for moisture sensitive products. It directly influences pharmaceutical quality, safety, and efficacy, controlling how tablets interact with its environment before and after administration. The ability to quantitatively assess coating integrity, uniformity, and moisture barrier performance is essential for controlling critical process parameters (CPPs). These are present at every stage of formulation and manufacturing, controlling them ensures that variability in the process does not compromise product performance. This application note demonstrates the use of Dynamic Vapor Sorption (DVS) as a Process Analytical Technology (PAT) tool for evaluating tablet film coatings Opadry AMB and Opadry AMB II. *By measuring water vapor sorption isotherms, sorption kinetics, and diffusion coefficients, the DVS provides direct, quantitative insight into the moisture protection produced by different coating formulations and process conditions.*

Introduction

Opadry AMB and Opadry AMB II are two widely used film-coatings developed by Colorcon, Inc. for pharmaceutical solid oral dosage forms, each serving different functional purposes in drug formulation. Both coatings are specifically engineered to provide enhanced moisture-barrier protection for moisture-sensitive active pharmaceutical ingredients (APIs). The coating is based on polyvinyl alcohol (PVA), a film forming polymer whose semi crystalline structure and intermolecular hydrogen bonding network restrict free-volume transport pathways, resulting in reduced water vapor permeability and a slow moisture diffusion to the tablet core (1). The Opadry AMB II formulation has been optimized to be PEG-free, improving both the moisture resistance and the chemical stability of the coated product. However, studies have shown that despite these barrier enhancements, PVA-based films remain sensitive to humidity and temperature fluctuations (2). When exposed to elevated relative humidity, PVA coatings can undergo a glassy-to-rubbery transition, which increases polymer chain mobility and consequently enhances water absorption. The moisture-barrier characteristics of both systems are governed not only by formulation composition but also by coating microstructure, thickness, and processing conditions, which together determine the effective diffusion rate of water vapor through the film and, consequently, the level of protection of the tablet core.

Traditional approaches for determining moisture uptake to assess coating quality include long term studies, Karl Fischer titration, or weight gain measurements. These experiments are time consuming and often lack the sensitivity needed to differentiate subtle changes in coating structure or process conditions. As a result, there is increasing demand for efficient, informative analytical techniques that can be implemented during drug development. To address these limitations, Process Analytical Technology (PAT) testing tools are used for assessing coating performance by providing sensitive, quantitative insight into moisture interactions. The DVS can be used as a gravimetric

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technique that measures the interaction of materials with water vapor under precisely controlled temperature and relative humidity (RH). When applied to coated tablets, DVS enables direct measurement of moisture uptake, sorption kinetics, and diffusion behaviour, making it a highly effective PAT tool for evaluating coating performance and robustness.

This application note presents results from the analysis of four distinct coating trials conducted at Colorcon, West Point, Pennsylvania, designed to evaluate the impact of coating process conditions on the quality of film-coated placebo tablets. The study systematically compared wet versus dry coating processes and formulations applied with and without sub-coats. Key process parameters, including atomizing air pressure, pattern air pressure, pan speed, and process airflow, were deliberately varied to understand their influence on coating performance. The resulting coated tablets were characterized using Dynamic Vapor Sorption (DVS) to assess moisture uptake and barrier performance. Quantitative videoscope imaging was used to observe coating coverage, uniformity, and surface quality.

Methods

Moisture Sorption

Moisture sorption behaviour was characterized using a Dynamic Vapor Sorption instrument at Surface Measurement Systems in combination with Karl Fischer moisture analysis. Vapor sorption kinetics and equilibrium isotherms were also measured using the DVS. Sorption and desorption behaviour were determined gravimetrically using an ultra-microbalance with a mass resolution of ±0.1 μg, enabling the detection of very small changes in sample mass associated with vapor uptake and loss. The entire measurement system was contained within a temperature-controlled incubator, maintaining the experimental temperature to within ±0.1 °C.

Samples were placed in a mesh sample pan designed to allow unrestricted vapor access to all surfaces of the tablet. Prior to sorption measurements, samples were dried at 0% relative humidity (RH) at the target temperature until a stable dry mass was achieved. Following equilibration at dry conditions, samples were exposed to 60% RH, and mass changes were continuously recorded until equilibrium was reached.

To complement gravimetric sorption data, moisture content and weight gain was determined using a Mettler-Toledo DL38 Karl Fischer titrator equipped with a homogenizer, in conjunction with a Mettler-Toledo XS205 dual-range analytical balance. Tablets were stored under accelerated conditions (40°C / 75% RH) for two months prior to analysis to quantify moisture uptake and assess hygroscopic behaviour.

Coating Thickness

Coating thickness was evaluated by cross-sectional imaging. Tablets were fractured to expose the coating cross section, and measurements were obtained using a Keyence VHX-600 digital microscope with interchangeable lenses providing magnifications from 0–20X up to 1000–5000X.

High-resolution three-dimensional images were acquired using a 54-megapixel 3CCD camera system, providing enhanced depth of field compared with conventional optical microscopy. This imaging capability enabled accurate thickness determination across the coating layer. At least 20 measurements per region were collected from randomly selected tablets.

Results

Moisture Sorption Isotherms and Kinetics

Dynamic vapor sorption isotherms were generated for core tablets, control tablets (C10056), and coated tablets prepared using Opadry AMB and Opadry II coating formulations. A description of the coating compositions and processing conditions is provided in Table 1. The mass change profiles and comparative isotherms are shown in Figures 1 and 2.

The DVS data presents clear differences in moisture uptake behavior. Core tablets and Opadry AMB coated tablets exhibited similar sorption profiles, with significantly higher equilibrium mass gains compared to tablets coated with Opadry II. This suggests that the Opadry AMB coating provides less effective moisture barrier performance compared to Opadry II.

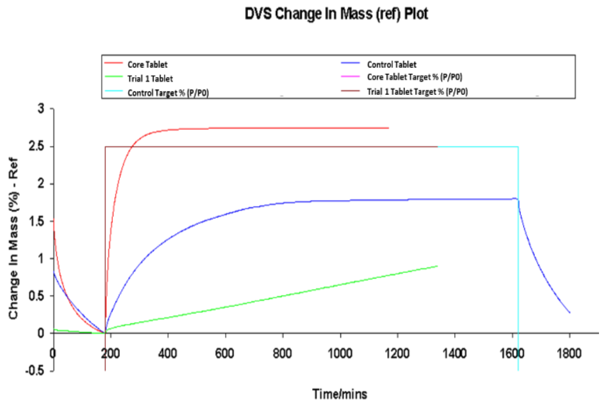

Figure 1. Sorption isotherms for Control, Core and Trial 1 tablets

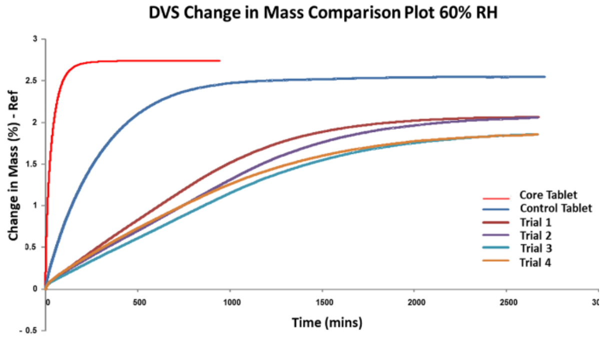

Figure 2. Sorption isotherms for Control and Trial 1-4 tablets

In trials 1 and 2, Opadry II was only used as a top-coat on top of an Opadry AMB layer. This showed higher overall changes in mass compared to trials 3 and 4, where Opadry II was used throughout the full coating process. These differences are evident in both the kinetic uptake curves and the equilibrium plateaus observed at 60% RH (Figure 2), indicating that thickness and extent of coverage of Opadry II layer are key factors governing moisture transport.

Table 1. Tablet Coating and mass uptake

Sample	Coating Material	Solid Content
C10056	Wet AMB pink	4% wt gain of 21% solid
	Top Coat Opadry Clear	1% wt gain of 7 % solids
Trial 1 AMB 50°C	Wet AMB pink	4% wt gain of 20% solids
	Top Coat Opadry II Clear	0.5% wt gain of 7.5 % solids
Trial 2 AMB 56°C	Dry AMB pink	4% wt gain of 20% solids
	Top Coat Opadry II Clear	0.5% wt gain of 7.5 % solids
Trial 3	Seal Coat Opadry II Clear	1.0% wt gain of 7.5 % solids
	AMB Pink	4% wt gain of 20% solid
	Top Coat Opadry II Clear	0.5% wt gain of 7.5 % solids
Trial 4	Seal Coat Opadry II Clear	1.0% wt gain of 7.5 % solids
	Opadry II Pink	4% wt gain of 20% solid
	Top Coat Opadry II Clear	0.5% wt gain of 7.5 % solids

Effect of Coating Process and Thickness

For Trial 2, tablets were dried in the coating pan prior to starting the coating process. These tablets were observed to be more sensitive to environmental humidity changes, as shown by higher mass gains in the DVS experiments. The gravimetric response (Table 2) of Trial 2 had higher uptake compared to Trials 3 and 4, suggesting reduced barrier performance due to thinner coating. Cross-sectional videoscope images (Figures 3 and 4) confirm that Trial 2 tablets possessed a thinner overall coating compared to the other Opadry II based trials. The reduced coating thickness layer explains the increased moisture uptake observed in the DVS data, highlighting the importance of both coating formulation and process conditions in achieving effective moisture protection.

Table 2. Tablet Coating and mass uptake :

Sample	Weight Gain 40°C 75%RH (%)	Moisture (%)	Δ in Mass (%)	Average Coating Thickness (µm)	Diffusion Coefficient (cm^2/sec)
Core tablets	0.88	4.77	2.75	N/A	N/A
C10056	0.67	4.56	2.50	37.15	3.63×10^{-10}
Trial 1	0.68	4.30	1.90	33.61	1.14×10^{-10}
Trial 2	0.80	4.37	1.75	27.66	7.02×10^{-11}
Trial 3	0.67	4.34	1.55	33.79	6.50×10^{-11}
Trial 4	0.58	4.31	1.60	38.31	1.22×10^{-10}

Moisture Content and Uptake under Controlled Temperature and Humidity Conditions

Moisture content and weight gain following storage at 40°C / 75% RH for two months are summarized in Table 2 and compared with the DVS results. The total moisture content of Trials 1 to 4 was broadly comparable across the Opadry II based formulations. In contrast, the control tablets (C10056) and Core tablets showed higher moisture contents, consistent with their higher equilibrium mass gains observed in the DVS isotherms. Weight gain measurements followed a similar trend. Tablets coated entirely with Opadry II (Trials 3 and 4) showed lower mass increase compared to tablets coated with Opadry AMB or partial Opadry II sub-coats. This further supports better moisture barrier performance of the Opadry II coating system.

Figure 3. Videoscope images of a whole tablet, cross sectional area of the tablet and the tablet under Trial 1 conditions

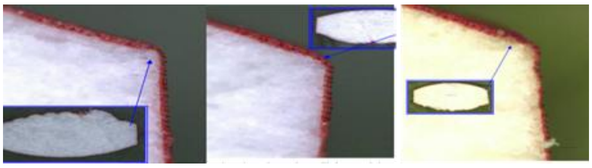

Figure 4. Coating thickness of the tablet under conditions of Trial 2, Trial 3 and Trial 4

Water Vapor Diffusion Analysis

The initial stages of vapor uptake following each step change in relative humidity were used to calculate water vapor diffusion coefficients within the coating films (3). For a thin film of thickness d exposed to a step change in humidity, the early-time sorption behavior for a two-sided film is described by Equation (2):

$$\frac{M_t}{M_\infty} = \frac{4}{d}\sqrt{\frac{Dt}{\pi}}$$

Where Mt is the amount adsorbed at time t, $M\infty$ is the amount adsorbed at thermodynamic equilibrium, and D is the diffusion constant. For values of Mt/M∞ <0.4, a plot of Mt/M∞ versus t1/2/d is linear, allowing the diffusion constant D to be calculated from the slope. In these calculations, film thickness obtained from cross-sectional imaging was the only required input parameter. To accurately monitor fast diffusion processes, mass data were recorded every 2 seconds. Excellent linearity was obtained for all diffusion plots, with minimum R^2 values of 99.9%, confirming the reliability of the diffusion analysis.

The calculated diffusion coefficients (Table 2) further differentiate the moisture barrier performance of the coating systems (4). When comparing tablets with similar coating thicknesses, Opadry II consistently exhibited lower diffusion constants than Opadry AMB, indicating slower water vapor transport through the film. For example, Trial 4 showed a lower diffusion coefficient than the control C10056 (1.22×10^{-10}

cm^2/s versus 3.63×10^{-10} cm^2/s, respectively). Similarly, Trial 3 exhibited a lower diffusion coefficient than Trial 1 (6.50×10^{-11} cm^2/s versus 1.14×10^{-10} cm^2/s).

Conclusion

The DVS isotherms, moisture content measurements, coating thickness analysis, and diffusion constant calculations consistently demonstrate that tablets coated with Opadry II provide superior moisture protection compared to those coated with Opadry AMB. When considered collectively, the data show that coating thickness and the extent to which the coating system is applied throughout the process, along with the type of coating are key parameters governing moisture uptake and transport. The sensitivity of the DVS to changes in coating composition, thickness, and processing conditions allowed correlations to be made between coating design and functional performance. This study highlights the value of DVS as a PAT tool for improving the understanding of pharmaceutical tablet coating behavior. By providing quantitative insight to moisture sorption kinetics and diffusion processes, the DVS can be effectively used during development to define design space, identify critical process parameters, and support the selection and optimization of coating systems to ensure efficient moisture protection.

References

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