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DVS Application Note 107

DVS Endeavour: An Introduction to the Endeavour System and its Applications

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This application note will introduce the DVS Endeavour system and a wide range of applications it can be applied to. This multi-balance system can be used to investigate different samples with different uptake properties or measure the uptake of 5 different loadings of the same sample.

Introduction

The DVS Endeavour has 5 balances in isolated sample chambers, allowing simultaneous parallel sorption analysis (Figure 1).

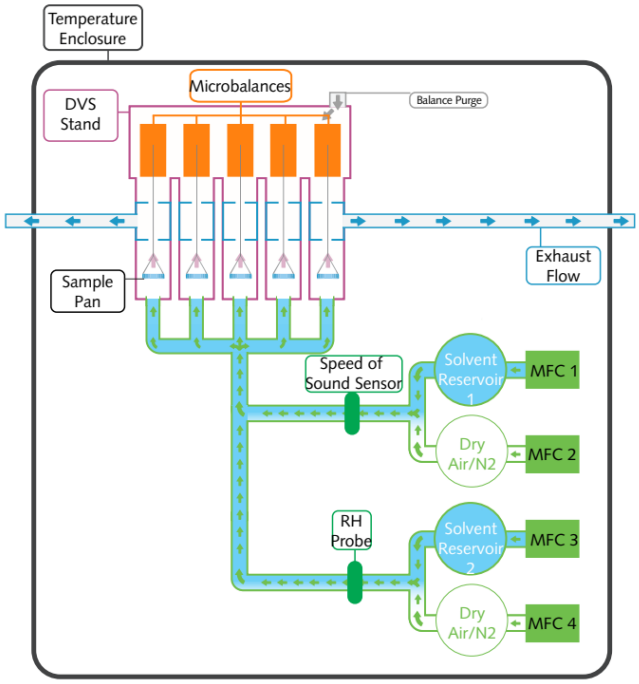

Figure 1 *Schematic of the DVS Endeavour*[1]

For high fidelity results, five of the same sample can be measured at the same time with exceptional reproducibility as seen in Figure 2 for five samples of Avicel. This is further exemplified with identical isotherms shown in Figure 3, showing a total sorption capacity of 15.55% w/w with good reproducibility.

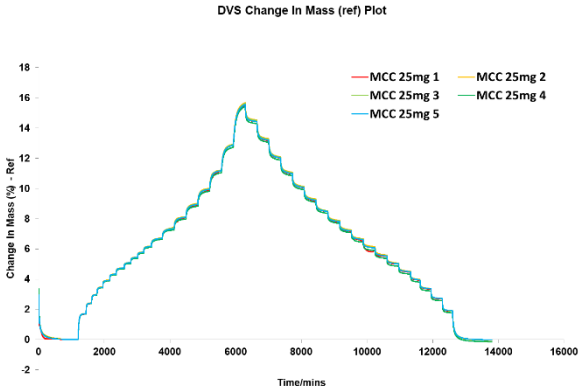

Figure 2 *Avicel water sorption kinetics, measured using the DVS Endeavour at 25°C*

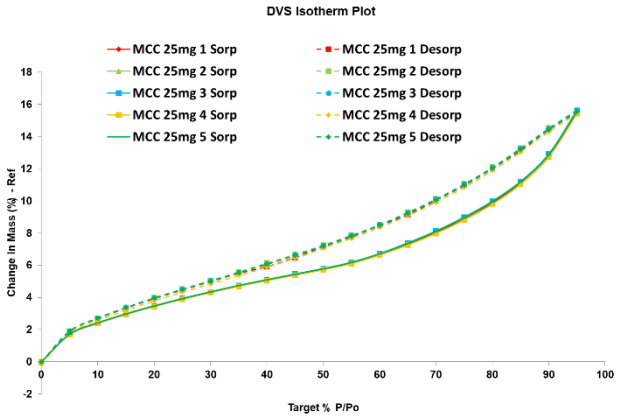

Figure 3 *Five Avicel water sorption isotherms measured in parallel using the DVS Endeavour at 25°C*

Five different samples can also be loaded for high throughput experiments. Figure 4 shows water sorption data for five different fibres: bleached hair, virgin hair, cotton, polypropylene and polyester. While each fibre has slightly different kinetics, the next relative humidity stage will not proceed until all the samples have reached an equilibrium mass. This ensures that all samples are allowed to reach equilibrium so that all samples have maximised their uptake at a particular RH or P/P_0. Five experiments took almost 5,000 minutes to complete, but it would have taken 25,000 minutes to complete on a single balance system.

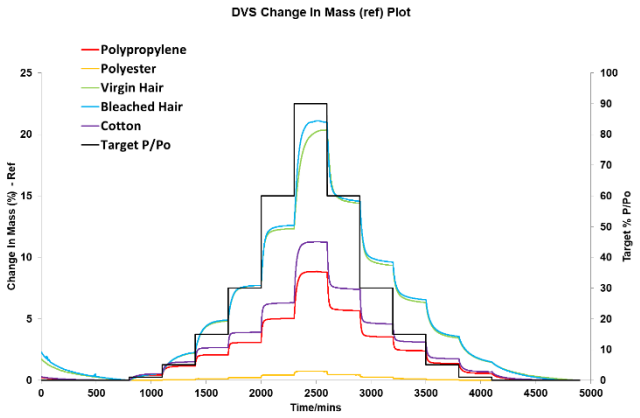

Figure 4 *Five different fibre sample water sorption kinetics measured at the same time using the DVS Endeavour at 25°C. Humidity generation steps will not proceed until all the samples have reached a mass equilibrium.*

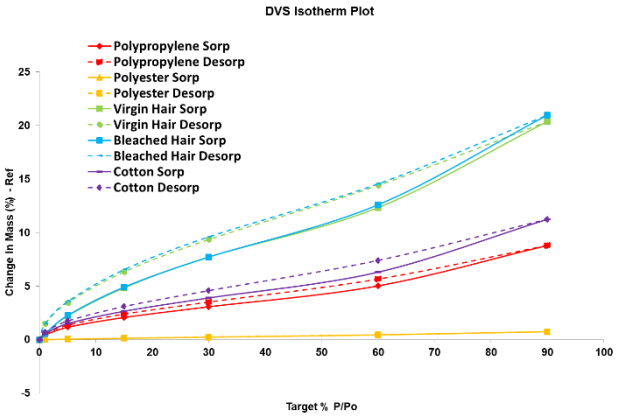

Figure 5 *Water sorption isotherms for five different samples measured at 25°C using the DVS Endeavour.*

The isotherms for these fibre samples can be found in Figure , demonstrating the high hydrophilicity of the hair samples and hydrophobicity of the polyester sample. This establishes the usefulness of using the DVS Endeavour as a high-throughput method for screening properties of different materials.

DVS Endeavour Specifications and accessories

Table 1 shows a comparison of the specifications for the DVS Resolution and DVS Endeavour.[1]

Table 1 *A comparison of the specifications for the DVS Resolution and the DVS Endeavour*

	DVS Resolution	DVS Endeavour
Number of Balances	1	5
Temperature Range	5°C to 85°C	10°C to 70°C
Temperature Stability	±0.05°C over 6 hours	±0.05°C over 6 hours
Humidity Range at 25°C	0-98% RH	0-98% RH
Humidity Stability	±0.1%	±0.1%
Gas Flow	200 sccm	400 sccm

The DVS Endeavour can be configured in Aqua or Standard configurations. The DVS Endeavour Aqua system is a water-only system equipped with a capacitance humidity probe, while the DVS Endeavour Standard system is capable of organic vapour sorption with the addition of one Speed of Sound sensor. The standard configuration also allows co-adsorption to be measured, with water and organic vapour simultaneously.

Similar to the DVS Resolution, the DVS Endeavour can be configured with an SMS ultra-balance low mass version which measures samples between 1 and 1000 mg, or a SMS ultra-balance high mass balance which measures samples between 10 and 5000 mg.

The DVS Endeavour can be equipped with up to five preheaters and one camera accessories as an option.

DVS Endeavour Case Studies

i) Amorphous vs Crystalline Lactose Water Sorption

Figure 6 shows the water sorption kinetics for five amorphous lactose samples from the same batch. Using the DVS Endeavour five samples can be measured at the same time showing incredibly similar behaviour. In the first cycle, there is a drop in mass at 90%RH stage characteristic of an amorphous to crystalline transition. Water is retained in the second drying stage, which may be water trapped through the crystallisation process.

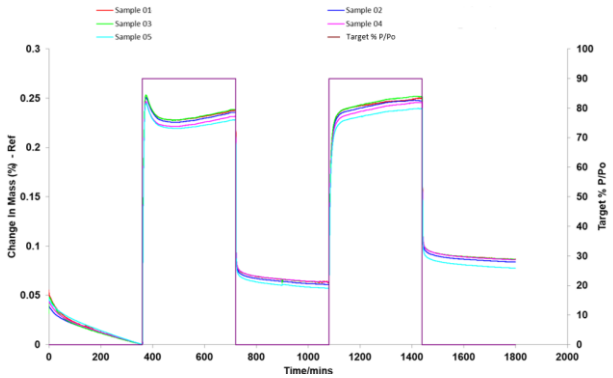

Figure 6 *Water sorption kinetics for five amorphous lactose samples at 25°C, using the DVS Endeavour.*

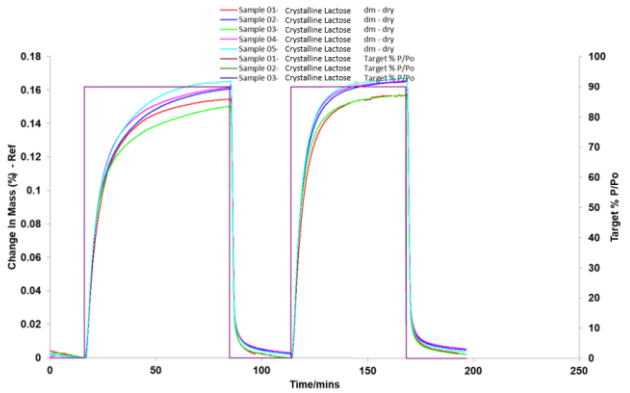

Figure 7 *Water sorption kinetics for five samples of fully crystalline lactose at 25°C.*

Amorphous lactose is known to (partially) crystallize to the monohydrate form.

In the second cycle of exposure to 90%RH there is no drop in mass, indicating that all the amorphous content of the samples has been fully crystallised in the first cycle.

The same experiments were repeated for samples of fully crystalline lactose with the kinetic data shown in Figure 7. There is a clear difference in the data for the crystalline samples, with no drops in mass as seen in Figure 6 for the amorphous samples. This indicates that the lactose is indeed fully crystalline and does not go through a phase transition. For crystalline lactose, water sorption is fully reversible for the two water sorption cycles.

Figure 8 shows the data for the amorphous lactose experiments and the crystalline lactose experiments. What is immediately clear is the difference in kinetics for the water sorption processes; crystalline lactose reaches an equilibrium significantly faster than the amorphous samples. This is due to crystalline lactose being limited to adsorbing water on the surface of the crystalline structure[2]. This also accounts for the reduced sorption capacity of the crystalline sample in comparison to the amorphous sample which can sorb into the bulk of the material as well as the surface.

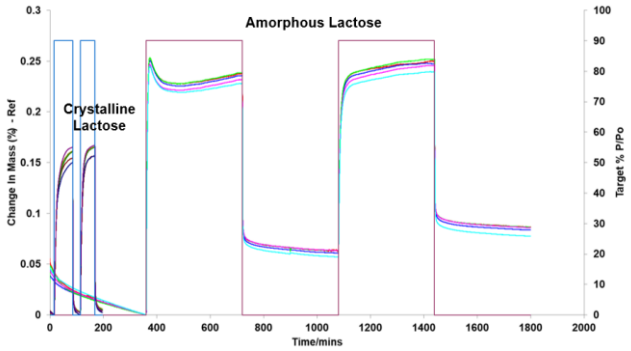

Figure 8 *Comparison of the water sorption kinetics of crystalline and amorphous lactose samples, measured by the DVS Endeavour at 25°C.*

ii) API Crystallisation using Ethanol

Recrystallisation kinetics can be measured for Active Pharmaceutical Ingredients (API's) using the DVS Endeavour. Figure 9 demonstrates the recrystallisation of 5 samples of an API using ethanol at 25°C. After initial drying for 20 minutes, the samples are exposed to 70% P/P_o ethanol, where a mass increase is followed by the characteristic mass decrease associated with a crystallisation phase change. As observed for amorphous lactose in Figure 6, some ethanol is retained by the API during the desorption stage having been incorporated into the crystal structure.

In the second cycle a slight mass decrease is observed indicating further crystallisation processes.

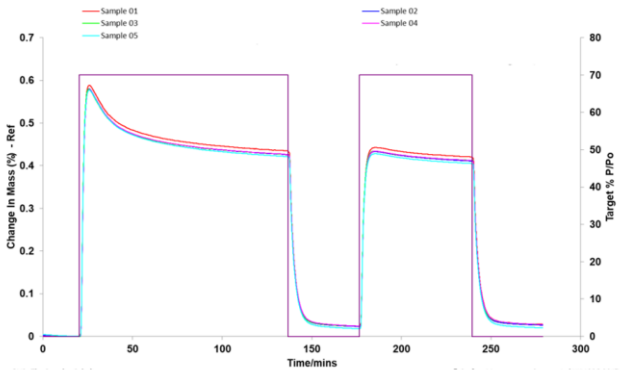

Figure 9 *The crystallisation of five samples of an API measured using ethanol at 25°C.*

iii) Amorphous Content determination

In addition to measuring crystallisation kinetics of amorphous samples, the amorphous content of the sample can also be measured using a straightforward DVS experimental method established by Mackin *et al*.[3] Figure 10 shows the amorphous content determination for five amorphous lactose samples, as measured by the DVS Endeavour.

The experimental procedure is performed as follows:

1. Initial drying of the samples under 0%RH at 25°C for 10 hours.
2. The samples are exposed to a partial pressure of 20% RH, where lactose is not expected to crystallise. Here there is both amorphous and crystalline lactose present.
3. Samples are then exposed to 95%RH to induce crystallisation in the amorphous phase.
4. The samples are dried at 0%RH.
5. Finally, the samples are exposed to 20%RH for a second time, where it is expected that the samples are fully crystalline. This stage will have a lower uptake due to the absence of amorphous lactose.

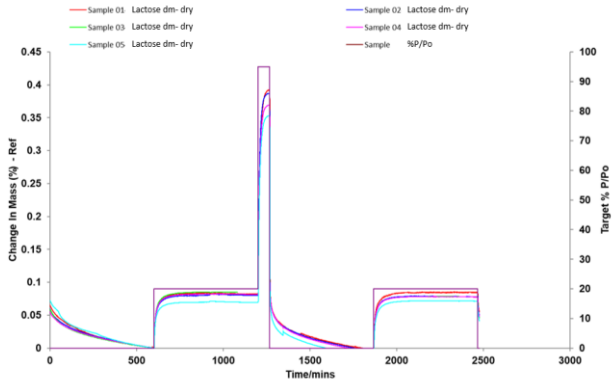

Figure 10 *The determination of amorphous content in five lactose samples, using water to crystallise the samples. All samples were measured using the DVS Endeavour at 25°C.*

The amorphous content of the samples can be calculated by the differences between the first sorption stage at 20%RH and second stage at 20%RH. The difference is directly associated with the amorphous content in the samples. Figure 10 demonstrates how the five samples of the same amorphous lactose batch have comparable amorphous contents as measured by this method.[3]

Conclusion

The DVS Endeavour provides high-throughput and high-fidelity measurements due to its five-balance system. This application note has demonstrated the high reproducibility of measurements across each balance, and a range of experimental methods that can be performed using the DVS Endeavour. Further information can be found in the DVS Endeavour Brochure.

References

1. DVS Endeavour Brochure, Surface Measurement Systems
2. A. Newman, G. Zografi, Journal of Pharmaceutical Sciences 108 (2019) 1061-1080
3. Mackin, L., Zanon, R., Park, J. M., Foster, K., Opalenik, H., Demonte, M. Int. J. Pharm. 2002; 231, 2: 227-236

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