Dynamic Vapor Sorption Sampling Considerations

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INTRODUCTION

Dynamic vapor sorption (DVS) evaluates the water adsorption / desorption behavior of materials as a function of relative humidity and temperature. Since this behavior can be affected by previous sample history as well as sample form, it is important to consider those variables when designing and interpreting DVS experiments.

EXPERIMENTAL

Most materials over time reach equilibrium with the relative humidity of their environment. Hence, DVS results obtained by directly running materials "as received" are most useful for qualitative interpretations / comparisons. If it is important to accurately quantify water gains / losses (e.g., to verify formation of hydrates), the material must be brought to a known "starting point" prior to the DVS experiment. This is generally accomplished by drying at 0 %RH and modest temperature until a constant weight is obtained. Care must be taken, however, to ensure that the material's original structure is not altered during drying. Since water adsorption and diffusion into materials is typically a slow process, sample form (e.g., powder versus a solid tablet) can also affect results. Although largely a surface area effect, other factors such as pore dimensions in the structure of solids can also be involved.

RESULTS and DISCUSSION

Figure 1 compares DVS results for the anti-inflammatory drug, diclofenac "as received" and after drying at 60 °C and 0 %RH for 4 hours. The general shape and level of water adsorption / desorption are the same with and without drying, indicating that the drying conditions chosen, while sufficient to remove 1-2 % of adsorbed surface water, do not alter the material's structure or subsequent water adsorption / desorption properties. The DVS curves indicate that diclofenac initially forms a hydrate at higher humidities, which dehydrates as humidity is subsequently lowered. The second increasing humidity curve does not overlay the original curve implying a structure change during the hydration / dehydration process. Although the actual amount of water adsorbed / desorbed for the "as received" sample is qualitative, useful information is still obtained.

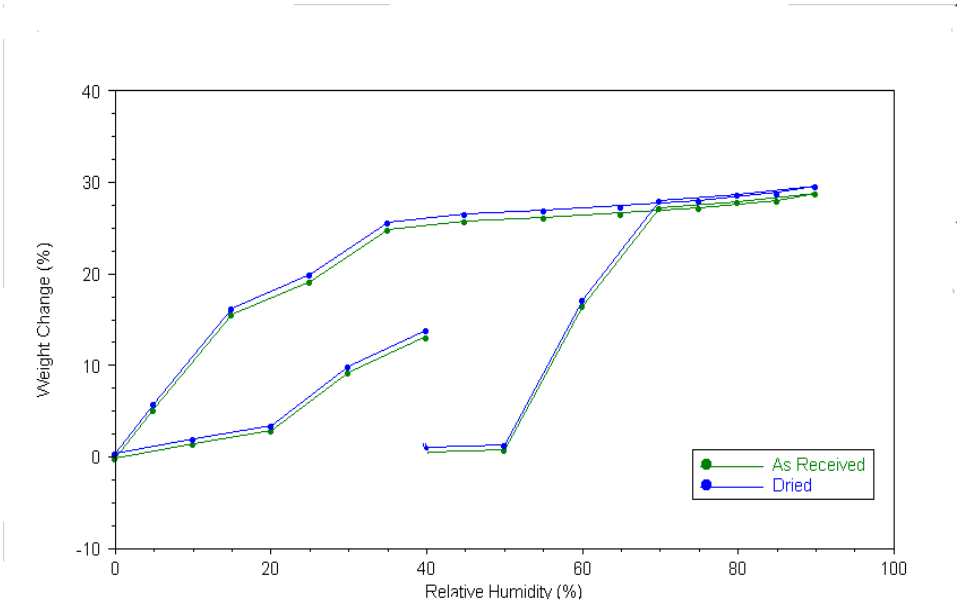

Figure 1. Comparison of Dried Versus "As Received" Dichlofenac

Table 2, on the other hand, represents a situation where accurate quantitation of the water adsorbed at different humidity levels is the primary measurement objective. Microcrystalline cellulose is a common pharmaceutical excipient often used to evaluate the performance of DVS instruments. The results clearly show that previous sample history ("as received" sample) and proper drying have a large effect on the weight changes observed at three different humidity levels versus the change expected based on another adsorption measurement technique. As expected, sample size, temperature, and time are important considerations during drying. Increasing temperature reduces the time required to completely dry the sample, whereas increasing sample size raises the time required for drying. If the sample is not properly dried prior to analysis, the weight gains observed, at each humidity level, are lower than expected because the "undried" material already contains some of the total water, which it can adsorb at the humidity levels being tested.

%RH	Weight Gain	Confidence Interval	Dried at 60C (10mg)	Dried at 25C (10mg)	Dried at 25C (2mg)	As received
11.1	2.13	+/-0.11	2.06	1.95	2.04	1.44
22.5	3.24	+/-0.12	3.25	3.12	3.24	2.67
33.0	4.15	+/-0.09	4.25	3.89	4.05	3.60

Table 2. Comparison of Microcrystalline Cellulose Results

Figure 2 illustrates the need for care when drying a material prior to DVS evaluation. Lactose monohydrate dried under mild conditions exhibits only a low level

of water adsorption indicative of surface adsorption on a crystalline structure. Lactose dried under more severe conditions however, exhibits different DVS behavior, reflecting dehydration during drying. The material rehydrates at 25 °C and humidity above 70 %RH. Once rehydrated, the subsequent DVS profile is consistent with the original monohydrate.

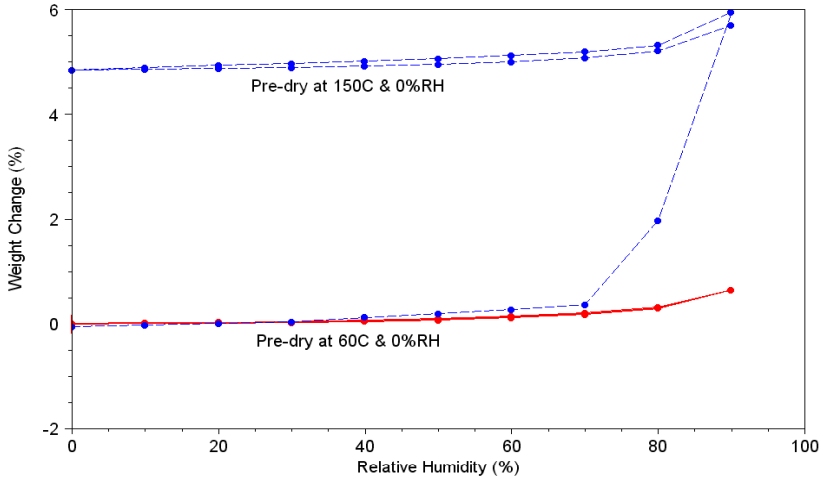

Figure 2. Effect of Drying Conditions on Lactose DVS Results

The DVS results (Figure 3) for tablets (with and without a surface treatment) show the expected slowing of water adsorption for the treated sample. Figure 4 compares the rate of adsorption for a similar whole uncoated tablet versus the tablet after powdering. The larger surface area after powdering results in more water adsorption in the time frame studied. This figure also shows that pan type can affect the results. Running the powder in mesh pans where the material is exposed on all sides to the humidified environment results in more rapid water adsorption.

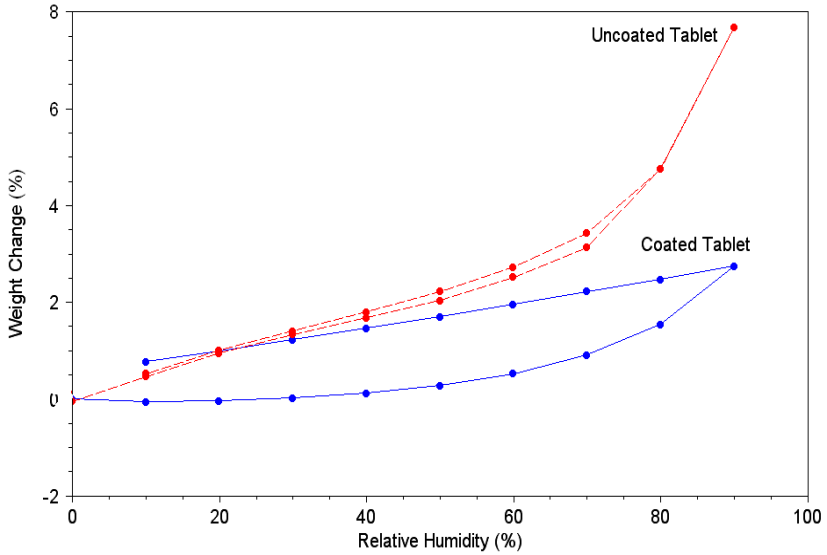

Figure 3. DVS Evaluation of Pharmaceutical Tablets

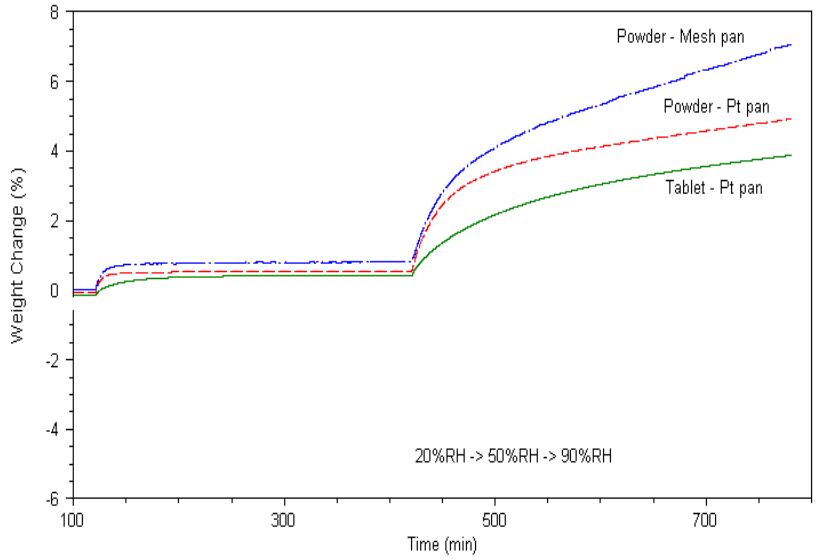

Figure 4. Sample Form & Pan Type Influence on DVS Results

Materials that readily adsorb water (hygroscopic materials) are difficult to evaluate since their weight changes continuously while trying to establish initial weight. This can be overcome by exposing separate samples of the material to each humidity of interest until equilibrium is obtained and then drying at 0 %RH to determine the total amount of water adsorbed. Figure 5 shows this approach for a hygroscopic peptide hydrate, which was too delicate to dry prior to analysis.

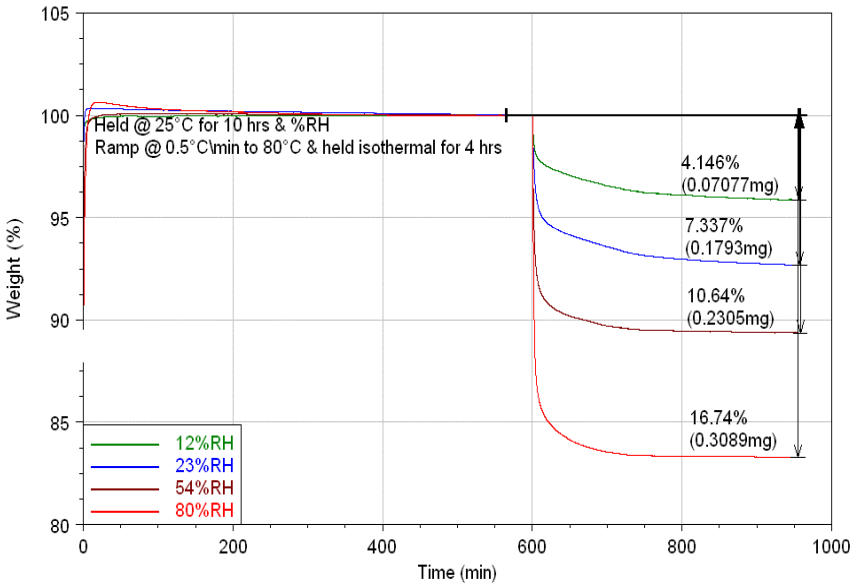

Figure 5. Peptide Hygroscopicity

Another method to eliminate concerns about hygroscopic materials changing while waiting for DVS analysis (particularly when using a DVS instrument with an autosampler) is to seal the samples in aluminum pans that are designed to be opened automatically just prior to the sorption analysis. Figure 6 illustrates this approach for polyvinylpyrrolidone (PVP). A lower than expected weight gain at 80 %RH for "pre-dried" PVP indicates that it equilibrates at roughly 6 % water pick-up after 8 hours at 25 °C and 25 %RH. However, sealing the "pre-dried" PVP in pans for 24 hours and then opening the pan for sorption evaluation, yields the expected result (42 +/- 2 % weight gain at 25 °C and 80 %RH). Sealed pans are the best choice for maintaining a known "as received" condition for materials prior to evaluation.

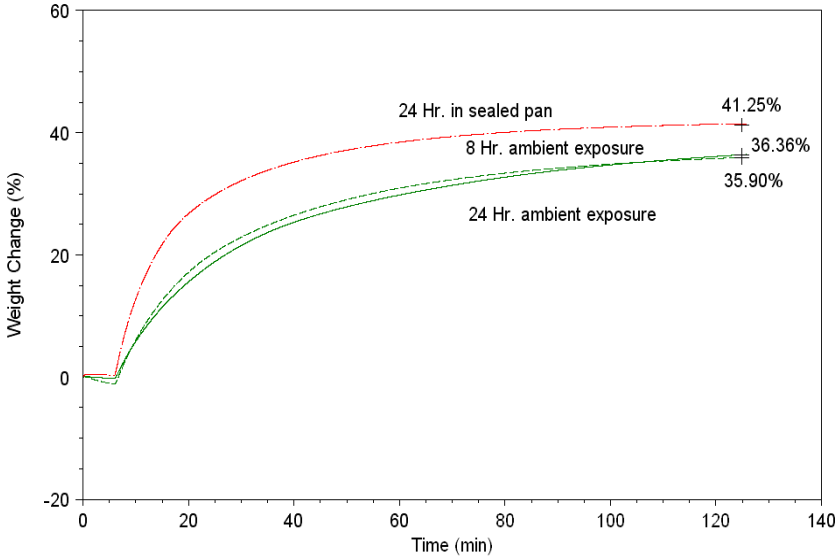

Figure 6. Evaluation of PVP Using Sealed Pans

SUMMARY

Like most analytical techniques, DVS results can be affected by many variables associated with sample handling and preparation prior to analysis. Hence, those variables must be considered in order to ensure best results.

KEY WORDS

DVS, Sampling conditions, Adsorption / Desorption, Relative humidity,

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