Characterization of Water Adsorption and Absorption in Pharmaceuticals

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INTRODUCTION

Dynamic vapor sorption (DVS) analysis can characterize material response to humidity changes. Typical responses are surface adsorption / desorption, bulk absorption, hydration / dehydration, and deliquescence. In surface adsorption, water can be weakly held to the surface by van der Waals forces (physisorption) or more strongly held (chemisorption). Physisorption is reversible by decreasing humidity or increasing temperature. Chemisorption is generally considered as irreversible. In bulk absorption, water is attracted deep into the internal structure of the material. Bulk absorption is reversible, but the kinetics are slower than for surface adsorption.

EXPERIMENTAL

Adsorption and absorption are evaluated by increasing humidity stepwise over a broad range, then decreasing it to the starting level and finally increasing it again to higher levels. The profiles are generated at constant temperature.

RESULTS and DISCUSSION

Figure 1 (carbamazepine) illustrates a typical surface adsorption profile.

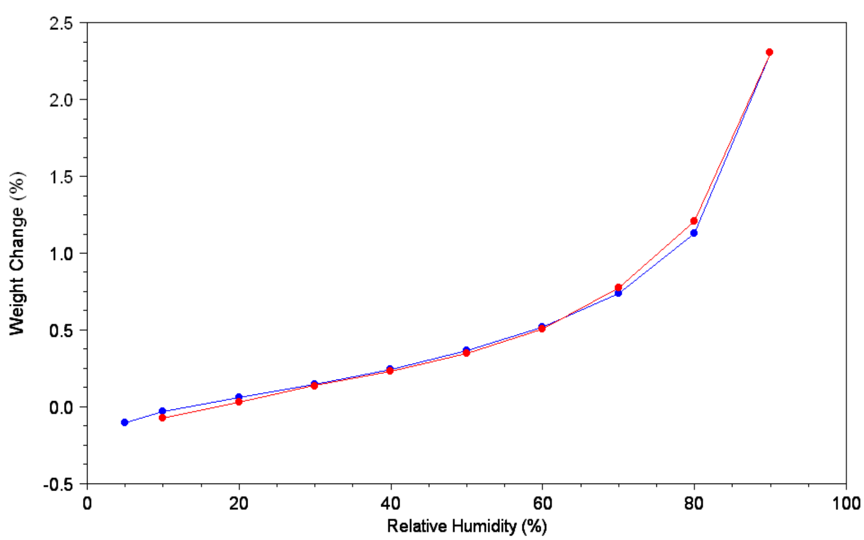

Figure 1. Carbamazepine Surface Adsorption

Surface adsorption is characterized by increasing / decreasing humidity curves, which coincide (i.e., overlap), as well as by small total weight changes over a broad humidity range. Crystalline materials generally exhibit this type of profile.

Bulk absorption profiles are shown in Figures 2 and 3. Choline (Figure 2) exhibits overlapping adsorption / desorption curves similar to that observed for surface adsorption. However, since the total water uptake is too large (150 % from 0-90 % RH) to be a surface phenomenon, bulk absorption into the material is occurring. As there is no evidence that the structure of choline changes as water enters and exits the material's internal structure, that structure must be very open

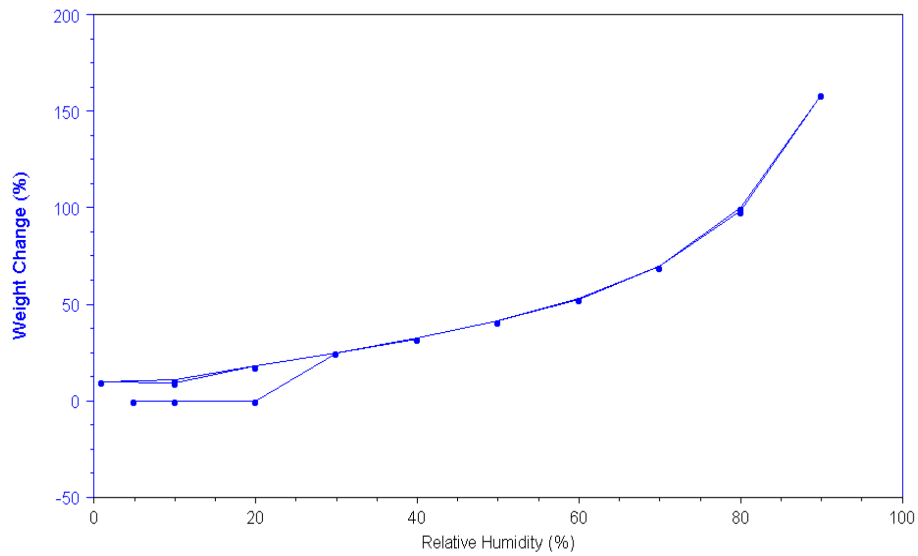

Figure 2. Choline Bulk Absorption

Partially amorphous, organic materials like starch (Figure 3) also show large water uptake indicative of bulk absorption. However, there is a slight but constant hysteresis (separation) between the water uptake and release curves, which probably reflects a slower diffusion of water back out of the starch's internal structure, which swells during water uptake.

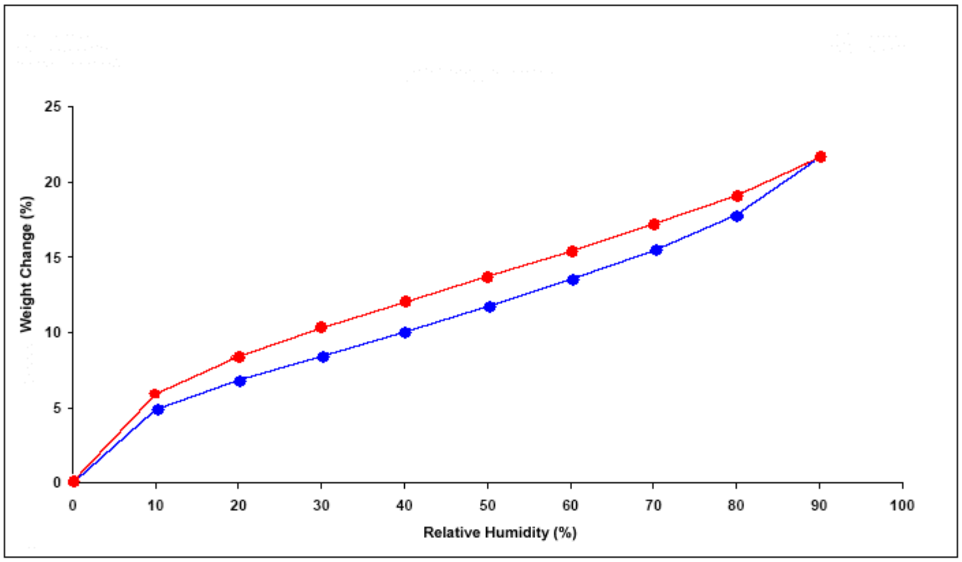

Figure 3. Starch Bulk Absorption

Materials, whose structures contain mesopores (internal cavities of 2-50 nanometers in diameter), show a rapid uptake of water (absorption) at higher humidities (Figure 4). The absorbed water results in capillary condensation, and the subsequent water release on decreasing humidity occurs more slowly resulting in hysteresis. The release occurs, however, over a narrow humidity range related to pore size.

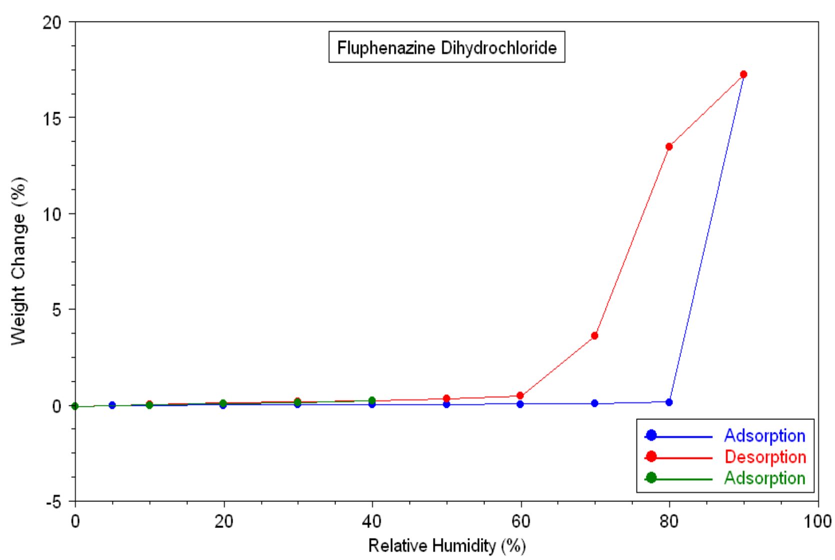

Figure 4. Absorption Into Mesopores

The previous examples all have reversible profiles indicating that the materials' structures are unchanged during water adsorption / desorption. Figure 5, on the other hand, shows a material whose morphology does change as bulk water absorption occurs. The slower release of absorbed water with decreasing humidity, and the associated significant hysteresis between the uptake and release curves over the entire humidity range, indicate the material has changed. Because there are no “step changes” in weight, the structural change is not the result of hydrate formation. The second increasing humidity curve follows the decreasing humidity profile as opposed to the original increasing humidity curve. Hence, the water-induced structural change is permanent. In addition, since the subsequent increasing and decreasing curves show no hysteresis, the new structure must be a fairly open one.

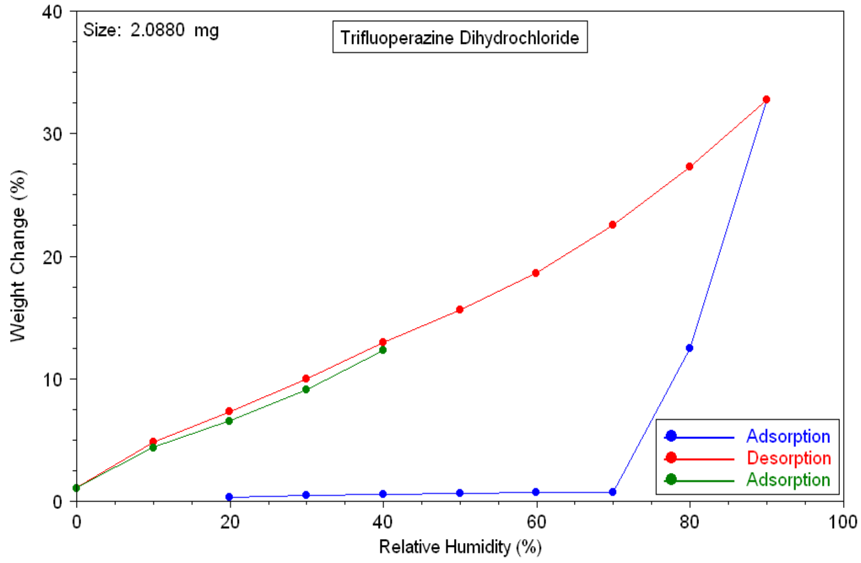

Figure 5. Bulk Absorption Induced Morphology Change

SUMMARY

The shape and amount of water adsorbed during a DVS adsorption / desorption profile provides insight into a material’s morphology. That information is valuable in projecting that material’s suitability for specific end-use applications.

KEY WORDS

Water Adsorption, Desorption, DVS, Bulk Adsorption, Hydration, Dehydration, Amorphous, Crystalline, Pharmaceuticals

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