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DVS Application Note 03

Polymorphism in Spray Dried Lactose

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The Dynamic Vapour Sorption(DVS) was used to study the physio-chemical behaviour of amorphous lactose. A humidity ramping method was used in the study.

Introduction

The presence of amorphous material in spray-dried powders such as lactose monohydrate is of great interest to the pharmaceutical industry, since even small amounts can lead to significant changes in the physico-chemical properties of the material. Recently DVS has been used to estimate the amorphous content of lactose powders for amorphous contents as low as 0.125% (compared to 10% detection limit for XRD) [1].This short application note describes the use of a DVS water sorption analyser to demonstrate the physico-chemical behaviour of a highly amorphous lactose powder by using a rapid humidity ramping method.

Method

A lactose powder with a high amorphous content was prepared by milling a small quantity of α-lactose monohydrate with a mortar and pestle. A small amount of sample (15mg) was then transferred to the DVS gravimetric moisture sorption analyser and dried under flowing nitrogen at 100 sccm and 25°C for 1 hour. The sample was then subjected to a constant shumidity ramp of 10% RH/hr from 0 to 100% RH at 25°C. The sample was then dried at 0% RH and a second ramping cycle was performed.

Results

Figure 1 shows the percentage change in mass of the sample as a function of relative humidity for both the first and second ramping cycles. The data shows a gross difference between the first and second cycle.

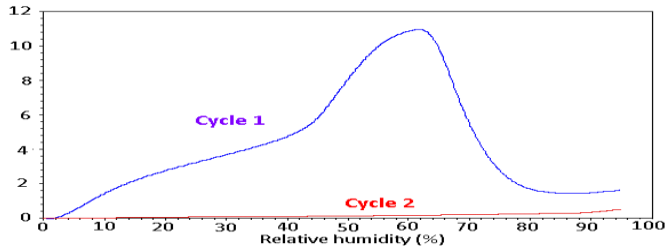

Figure 1. DVS humidity ramp experiment on highly amorphous lactose, at 25 C, ramp rate 10%/hr sample size 15 mg.

In the first cycle the sample takes up 11% by mass of moisture below 60% RH as would be expected for a highly amorphous material, however above 60% RH there is a sharp loss in mass to approximately 1.2% moisture content. Comparatively little uptake of moisture is observed in the second cycle, and the sample shows similar moisture sorption behaviour to α-lactose monohydrate.

Conclusion

The moisture behaviour of amorphous lactose is now well established [1]. Above a critical moisture content the glass transition temperature (T_g) of the amorphous regions is lowered to below 25°C such that the lactose molecules have sufficient mobility to recrystallise. After recrystallisation, the excess moisture is removed from the sample, resulting in an irreversible transformation

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References

[1] Buckton G. and Darcy P., Proc 1st Wld. Mt. APGI/APV, Budapest, 1995

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