

Moisture Stability of Powdered Milk Formulations

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This Application note describes the use of DVS technique for the stability study of powdered milk formulations.

Introduction

The moisture stability of powdered milk formulations is a crucial factor in ensuring the safe storage life of such products, particularly in countries that experience prolonged periods of both high humidities and temperatures. It is desirable that these products do not contain or release unbound moisture during storage, which under certain conditions may encourage growth of microbiological organisms.

Method

The moisture sorption properties of a powdered milk formulation were analysed using a DVS instrument. A sample size of 15mg was chosen as a suitable compromise between speed of analysis and homogeneity of the sample.

Results

Figure 1 shows the moisture sorption kinetics of the powdered milk formulation at 25°C measured by the DVS instrument. The data shows the change in mass as a percentage of the mass after drying under flowing nitrogen (blue line), and the humidity profile of the experiment (red line), as a function of time, for two complete sorption and desorption cycles. It is clear that the first sorption cycle shows dramatic differences from the second cycle, which can be attributed to irreversible moisture induced morphological changes in the sample. In the first cycle, at 60% RH, the sample undergoes a rapid loss in mass above a critical moisture content of about 60% which is consistent with the crystallisation of an unstable amorphous phase.

DVS

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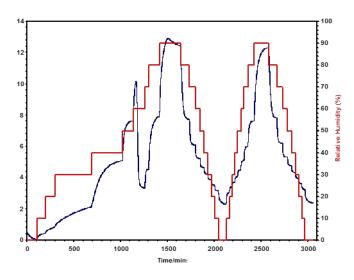


Figure 1. Moisture sorption kinetics for a powdered milk formulation.



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Indeed similar phenomena have been studied in detail by both DVS and differential scanning calorimetry (DSC) on spray dried amorphous lactose [1], where it has been shown that the moisture loss observed is due to the irreversible formation of a thermodynamically more stable crystalline form. The crystalline phase has a much lower affinity for moisture than the amorphous phase and hence the excess moisture is released as unbound or free moisture. In the case of the milk formulation this moisture loss is approximately 7% by weight at 60% RH. It is noted that the formulation contains 50% by mass of lactose. A further loss of about 0.5% at 90%RH is also observed, which may also be due to a crystallisation event. Figure 2 shows the sorption and desorption isotherms for both the first and second cycles and clearly demonstrates the irreversibility of the morphological transformations. The second cycle shows a very straightforward moisture sorption response with virtually no hysteresis.

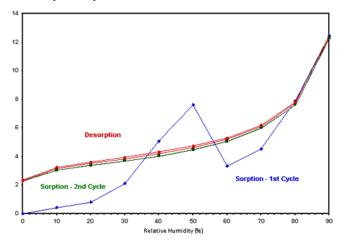


Figure 2. Sorption and desorption isotherms for a powdered milk formulation.

The isotherm data also shows that approximately 2.4% moisture is irreversibly bound to the sample after the first cycle, and this may either be due to formation of a crystalline hydrate, or encapsulation of moisture by the crystalline phase.

Conclusion

The DVS analysis therefore demonstrates that powdered milk formulations may undergo complex moisture induced morphological transformations, and that these may lead to the undesirable release of unbound moisture from the formulation.

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References

 G. Buckton and P. Darcy, The use of Gravimetric Studies to Assess the Degree of Crystallinity of Predominantly Crystalline Powders, Proc 1st Wld. Mt. APGI/APV, Budapest, 1995.

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