



*The water activity of salami is lower than that of uncured meat of similar water content due to the action of salt, which binds water*

## Using Moisture Sorption Isotherms in food engineering

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### Moisture

Water profoundly influences product attributes such as quality and safety. To completely understand water effects in a product requires an understanding of the amount of water (moisture content) that can be held at a given energy state (water activity). Moisture sorption isotherms describe the relationship between water activity and moisture content at a constant temperature. The nature of this relationship depends on the interaction between water and other ingredients. The amount of water vapour that can be absorbed by a product depends on its chemical composition, physical-chemical state, and physical structure. Consequently, the isotherm shape is unique to each product type due to differences in capillary, surface, and colligative effects.

### Measuring moisture sorption isotherms

Constructing an isotherm consists of collecting water activity and moisture content values over a range of water activities. The range of water activities used depends on the situation, but normally is 0.10  $a_w$  up to 0.90  $a_w$ .

In the traditional isotherm method, each point on the isotherm is determined by equilibrating a sample to a known water activity and then determining its equilibrium moisture content by weight. Typically, the sample is placed in a sealed chamber over a saturated salt slurry in excess. Different water activity levels are achieved by using different salts.

### Static Method

This traditional method has been automated by instruments programmed to automatically change the water activity of a sample in a stepwise progression. These instruments, often referred to as controlled atmosphere balances, utilise the 'Static' method. The instrument holds the sample at one water activity level until the sample weight stops changing, measures the water content by weight, and then dynamically moves to the next water activity. Automatic isotherm generators are much faster and less labour intensive than traditional desiccator methods. They also make it possible to conduct sorption kinetic studies. However, like traditional desiccator methods, 'static' instruments equilibrate the sample to a known water activity level. Since true equilibration between the sample and the vapour source requires an infinitely long period of time, they measure apparent equilibrium at the point when the change in sample weight is negligibly small.

### Dynamic Method

The Dynamic Dewpoint Isotherm (DDI) method directly measures water activity while gravimetrically tracking weight, so there is no dependence on equilibration to known water activity levels to determine water activity. Since the sample does not have to wait for equilibration to a specific water activity, this method is faster without sacrificing accuracy. It is simultaneously able to produce an unmatched number of data points.

## Uses for Moisture Sorption Isotherms

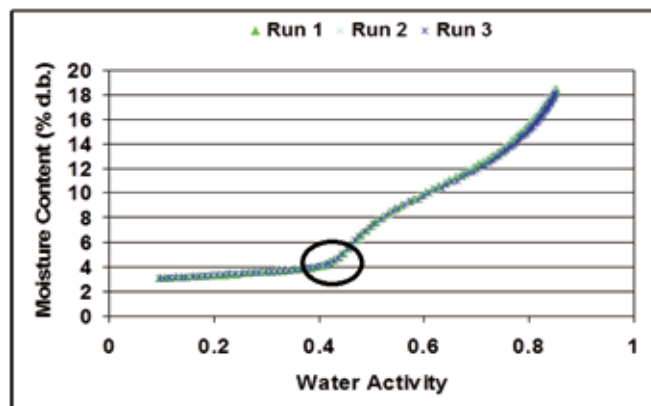
Moisture sorption isotherms are an important tool when formulating food to achieve specific qualities and attributes (Bell and Labuza, 2000). Despite their value, traditional isotherms have been limited by their low resolution. The high-resolution DDI method has opened up new and powerful possibilities. High resolution isotherms can reveal phase transition points – water activities at which products cake and clump, deliquesce, or go through glass transition. See Figure 1. for further explanation of this.

High-resolution dynamic isotherms can also be very helpful in:

- making shelf life calculations
- developing mixing models
- modelling temperature abuse
- determining the integrity of a protective coating or layer
- determining monolayer values
- making accurate packaging calculations.

## Phase Changes and Critical Water Activities

When high resolution isotherms are available, such as those produced using the DDI method, the shape of the isotherm can provide information about critical water activities and phase transitions. Sharp inflection points in the isotherm indicate phase transitions (equivalent to a glass transition) and can provide information about critical water activities for maintaining textural properties and preventing caking and clumping (Figure 1.). The exact inflection point in the curve, and hence the critical water activity, can be determined using second derivative curve smoothing strategies. Keep in mind that if data resolution of the



**Figure 1. Moisture sorption isotherm for spray dried milk powder at 25°C showing a phase change occurring at a critical water activity of 0.43**

isotherm is low, these inflection points cannot be identified. If the water activity of a product moves above the critical water activity for phase transition, the stability of the product will decrease as time dependent processes such as caking and crystallisation speed up significantly.

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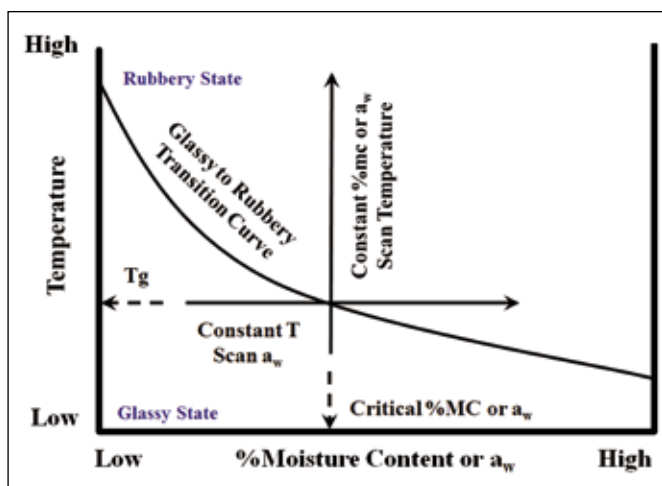


Figure 2. Strategies used to determine glassy to rubbery phase transitions in amorphous material. (Figure courtesy of Shelly Schmidt.)

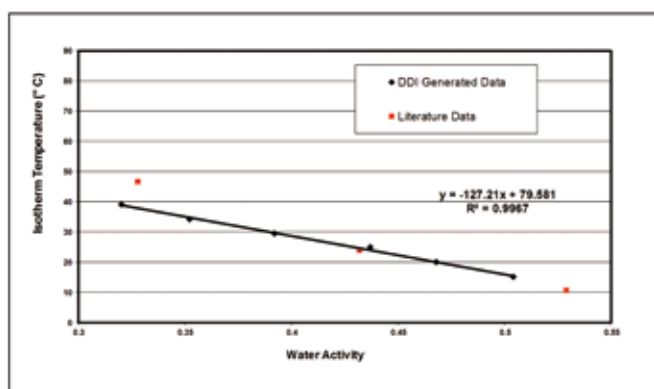


Figure 3. Water activity vs. isotherm temperature for spray dried milk powder showing a good linear relationship. Tg data taken from (Shrestha et al., 2007) shows good agreement to the isotherm data

Increasing temperature will cause the critical water activity to go down and can also result in loss of stability with no change in water activity. Determining phase transitions using isotherms is similar to determining glass transition temperature (Tg) with Differential Scanning Calorimetry (DSC), except that instead of holding water activity constant and scanning temperature, the isotherm analysis holds temperature constant and scans water activity (Figure 2). Consequently, the temperature of the isotherm when the critical water activity is determined should be similar to the Tg determined using DSC analysis for a sample at the critical water activity.

Graphing isotherm temperature and critical water activity for spray dried milk powder gives a good linear relationship with Tg values from Shrestha et al., (2007) at different water activities and agrees well with the data (Figure 3). This indicates that determining critical phase transitions using isotherm analysis is equivalent to determining them with DSC analysis. The linear relationship between isotherm

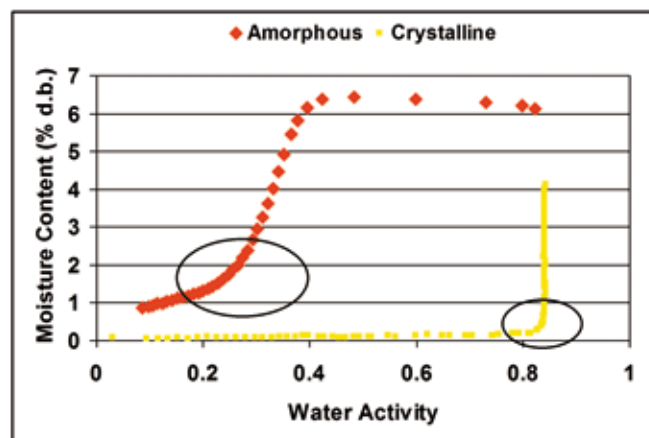


Figure 4. Moisture sorption isotherms at 25°C for crystalline (■) and amorphous (◆) sucrose showing a phase transition by the amorphous sucrose at 0.283 a<sub>w</sub> and the deliquescence of the crystalline sucrose at 0.815 a<sub>w</sub>.

temperature and water activity also makes it possible to predict the glass transition temperature at any water activity.

The moisture sorption isotherm can also illustrate differences between amorphous and crystalline material and provide information about the level of each in a product (Figure 4). Amorphous material is much more hygroscopic initially and then undergoes a phase transition, clearly visible with a DDI isotherm. Crystalline material has a very flat isotherm with large changes in water activity vs. moisture content until the deliquescence point is reached and the curve changes to vertical while waters of hydration are added. The deliquescence point, represented by a sharp inflection in the isotherm curve from horizontal to vertical, is easily identified with isotherms of high resolution without requiring visual inspection of the sample.

## Conclusion

Moisture sorption isotherms serve as a blueprint for moisture relations in foods and modern instrumentation has made it possible for anyone to analyse the moisture relations of their product. The efficacy of isotherms in food engineering depends on being able to achieve high data resolution without drastically increasing isotherm test time. New methods such as DDI make it possible to achieve the necessary resolution while reducing test times to days instead of weeks. These high resolution moisture sorption isotherms make it possible to model and engineer food products in ways not previously possible. The rewards are products that maximise safety, quality, and profitability.

## References

1. Bell, L.N., and T.P. Labuza. 2000. Moisture sorption: practical aspects of isotherm measurement and use. American Association of Cereal Chemists, St. Paul, MN.
2. Shrestha, A.K., T. Howes, B.P. Adhikari, and B.R. Bhandari. 2007. Water sorption and glass transition properties of spray dried lactose hydrolysed skim milk powder. *LWT - Food Science and Technology* 40:1593-1600.