

DVS Application Note 105

Shelf life Assessment of Meat Products by Dynamic Vapour Sorption

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Shelf-life assessment is of great importance to the food industry and companies are searching for new, rapid and easy ways to interpret techniques for shelf-life assessment. Dynamic Vapour Sorption (DVS) technique may be used to rapidly assess the sorption isotherms and the kinetics of moisture loss and drying of various materials in order to predict materials stability.

Introduction

Food and food ingredients can spoil due to a large number of factors, but the most common is microorganisms, including mould and bacteria. Some conditions, such as exposure to humidity can speed this process. Similarly, excessive drying may lead to materials instability as well as improper packing techniques which can cause the food products react with oxygen. Therefore, the moisture sorption properties of materials are critical for their shelf-life stability [1-3].

Food products and packaging materials have traditionally been evaluated by storing samples over saturated salt solutions of established relative humidities and then regularly weighed until equilibrium is reached [4]. However, the traditional methods have proved to be too inaccurate, labour intensive and take a long time to establish equilibrium.

Dynamic Vapour Sorption (DVS) technique may be used to rapidly assess the sorption isotherms and the kinetics of moisture loss and drying of various materials. The analysis of the experimental data may be performed using the Advanced Data Analysis add-in suite, facilitating rapid and reliable evaluation of the drying conditions.

This paper demonstrates the application of DVS equipped with a video microscope to characterise the stability of food products at different humidities and temperatures. DVS allows real time measurement of the water sorption kinetics over a wide range of relative humidities (from 0%RH to 98%RH) and temperatures (from 5°C to 85°C). The study will focus on meat products with intermediate shelf life and consider the changes in the moisture sorption isotherms of fresh and aged samples at different temperatures. This information may be used to help in the rational of food products as well design as recommendations for the packaging and storage conditions.

Method

The experiments were carried out on a DVS system equipped with a high mass microbalance capable of accommodating samples as heavy as 5g. The DVS instrument measures changes in mass which is related to uptake or release of sample at different concentrations and temperatures. The sample mass is monitored



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using an Ultrabalance with a resolution of \pm 1.0µg in a temperature controlled (\pm 0.1°C) incubator. The concentration of the moisture is controlled by mixing saturated vapour and dry air using mass flow controllers at a standard total gas flow of 200sccm.

Adsorption isotherms of cooked and smoked pork hot dogs were determined at 25°C and 35°C. One slice of the hot dog (27.4mg) was used to represent all the components of the product. The experiments were performed on fresh and two weeks old samples at different temperatures in order to demonstrate the differences in the moisture levels. Foer each experiment the sample was placed into a (large size aluminium) sampling balance pan and the starting %RH was set to 95% RH (P/Po, whereby P = Partial pressure of water vapour and Po = Equilibrium vapour pressure of water) decreasing to 0% RH using 10% RH decrements. The sample was then maintained at the final 0%RH step at 25°C for 360 minutes. The desorption cycle was followed by sorption from 0% RH to 95% RH using 10% RH increments. The weight change during the sorption and desorption cycles was then monitored, allowing for the hygroscopic nature of the sample to be determined. The %RH was maintained by the mixture of saturated water vapour and dry nitrogen (flow rate of 200 sccm). The percentage of mass change per minute (dm/dt) was set as 0.0002.

The desorption isotherm is found by placing an initially wet material under the same relative humidities, and measuring the loss in weight. Once the sample is dried adsorption isotherm is obtained by exposing the material to various atmospheres of increasing relative humidity and measuring the weight gain due to water uptake at a constant temperature. If the adsorption and desorption processes are not fully reversible, a distinction can be made between the adsorption and desorption isotherms bv determining whether the moisture levels within the product are increasing indicating wetting, or whether the moisture is gradually lowering reach to

equilibrium with its surroundings, implying that the product is being dried.

Moisture sorption isotherms of most foods are nonlinear, generally sigmoidal in shape. Foods rich in soluble components, such as sugars, however, have been found to show different isotherm characteristics. For interpretation purposes, the generalized moisture sorption isotherm for a hypothetical food system may be divided into three main regions, as detailed in Figure 1.

The monolayer region represents strongly bound enthalpy of vaporization water with an considerably higher than that of pure water. A typical case is sorption of water onto highly hydrophilic biopolymers such as proteins and polysaccharides. The moisture content theoretically, represents the adsorption of the first layer of water molecules. Usually, water molecules in this region may not be frozen and are not available for chemical reactions or as plasticizers. Most dried food products are empirically observed to display their greatest stability at moisture contents comparable to the monolayer moisture.



Figure 1. Generalized sorption isotherm for food products.

Heat of Sorption

The heat of sorption can be measured either by direct calorimetric methods or by applying the Clausius-Claypeyron equation at different temperatures using the DVS data. However,



calorimetric methods are less common because of the accuracy needed for precise measurement of the small quantities of heat evolved.

For this study the isoteric heat of sorption was calculated for a series of coverages using the isotherms collected at 25°C and 35°C.

Considering that foods are exposed to a range of temperatures during storage and processing and water activity changes with temperature, it is important to investigate the effect of temperature on the sorption isotherms.

Heat of sorption measurement would be useful because the heat of vaporization of sorbed water may increase to values above the heat of vaporization of pure water as the material is dehydrated to low moisture levels, which indicates that the energy of interaction between the sorbate and sorption sites is greater than the energy that holds the water molecules together in the liquid state. Therefore, the level of moisture content at which the differential heat of sorption approaches the heat of vaporization of pure water may be taken as indicative of the amount of 'bound' water existing in the material.

Surface adsorption should be considered when measuring the heat of sorption and the heat of sorption calculations have been based on the desorption branch of the isotherms. The experiments were performed on the as received wet samples by first measuring the desorption isotherms from 95% RH to 0% RH before measuring the sorption isotherms from 0% RH to 95 % RH.

Results

Figure 1 shows the net percent change in mass (based on dry mass, m₀) versus time for water sorption and desorption on the fresh and aged meat samples at 25 and 35°C. The fresh sample shows a higher initial moisture content than the aged sample and this is significantly more at 25°C. Both the fresh and aged products show different desorption behaviour at different temperatures. However, once dehydrated both products show





Figure 2. Moisture sorption kinetics showing the net percent change in mass (based on dry mass, m_0) versus time for the fresh sample at 25°C (green line) and 35°C (purple line) as well as the aged sample at 25°C (red line) and 35°C (blue line).



Figure 3. Moisture sorption isotherm plots for the fresh (a) and aged meat products (b) at 25°C.





Figure 4. Moisture sorption isotherm plots for the fresh (a) and aged meat (b) at 35°C.

Figures 3 and 4 show the net percent change in mass (based on dry mass, m₀) versus P/P₀ for water sorption (blue line) and desorption (red line) isotherms on the fresh and aged meat samples at 25°C and 35°C, respectively. Moisture sorption isotherms have an important role to play in the quantitative approach to the prediction of the shelf life of dried foods due to their sensitivity to moisture changes. The existence of hysteresis loops in the moisture sorption isotherms of food is indicative of a non-equilibrium state. Figures 3 and 4 show the characteristic hysteresis gap for food materials which remains over the entire partial pressure range. All samples show a narrower hysteresis gap at 35°C as at higher temperatures water molecules reach their activation energy and break away from their sorption sites more easily.

The increase in the equilibrium moisture content for the samples as the temperature increases is due to an increase in the molecular mobility of water which causes an increase in the amount of sorbed water.

Table 1. Water heat of sorption values over a range of surface coverages on the fresh and aged meat samples from isotherms at 25°C and 35°C.

Change in mass (%)	Heat of sorption (kJ/mol) Fresh Meat	Heat of sorption (kJ/mol) Aged Meat
9.0	-39.9	-32.1
17.0	-41.8	-32.4
26.0	-42.6	-32.1
36.0	-42.6	-30.6
45.0	-42.6	-29.2

Water heat of sorption values on the fresh and aged meat samples at 25°C and 35°C are shown in table 1. For the fresh sample the heat of sorption values are higher and increase with increasing coverage (change in mass), indicating that the interaction of water molecules in the vapour phase are increasing whereas the aged sample shows water molecules condensing on the surface.



Figure 5. Colour video images of the fresh (a) and aged (b) meat samples at 25°C and 95% RH.



From Figure 5, the in situ video images show changes in size and colour of the sample at different water concentrations. Condensation is present on and around the sample at 95% RH and the increase in the size of the white fatty tissue indicates a significantly higher uptake in the fatty tissue than in the red meat.

Conclusion

Heat of sorption values for the fresh meat sample increases with increasing coverage (change in mass), indicating that the interaction of water molecules in the vapour phase are increasing whereas the aged sample shows water molecules condensing on the surface. Once dehydrated both products show similar sorption behaviour at different temperatures.

There is no general equation for shelf life determination, but it is possible to predict the shelf life theoretically based on the moisture uptake of the material.

The information from focused experiments on fresh and aged food components can be used to predict storage stability under different conditions i.e. it should be possible to calculate the amounts of bound and free water that result in water activity and therefore affect food stability. Most foods have a water activity above 0.95 and that will provide sufficient moisture to support the growth of bacteria, yeasts, and mould. The amount of available moisture can be reduced to a point which will inhibit the growth of the organisms during storage.

The in situ video images show changes in size and colour of different components of the sample at different water concentrations and temperature.

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