Measuring Moisture Content Using Water Activity

Introduction

The terms moisture content and water content are often used interchangeably and represent a measure of the quantity of water in a product. Moisture content provides valuable information about yield and quantity, making it important from a financial standpoint. In addition, moisture content provides information about texture since increasing levels of moisture provide water mobility and lower the glass transition temperature.

Water activity represents the energy status of the water in the system. It is equal to the relative humidity of the air in equilibrium with a sample in a sealed chamber. It is defined as the vapor pressure of water in a sample divided by the vapor pressure of pure water at the sample temperature. Water activity provides valuable information about microbial spoilage, chemical stability, and physical stability. Water activity and moisture content together provide a complete moisture analysis.

Moisture content and water activity are currently measured using separate techniques or instruments. Water activity can be measured using either a capacitance or chilled mirror water activity instrument while moisture content can be measured using any one of the 35 different methods listed in Official Methods of the AOAC (AOAC, 1995). Combining the two analyses in one instrument can save time and labor. Decagon's new AquaLab Series 4 and AquaSorp Isotherm Generator now make it possible to measure both water activity and moisture content using Decagon's proven water activity measurement technology.

To measure moisture content using water activity requires an understanding of the relationship between the two parameters. This relationship, referred to as the moisture sorption isotherm, is complex and unique to each product type. It must be determined experimentally by measuring water content at several water activity values. This can be done manually with saturated salt slurries and desiccators or automatically using an isotherm generator instrument. Decagon's AquaSorp isotherm generator can rapidly generate robust isotherms with unmatched data resolution (Figure 1).

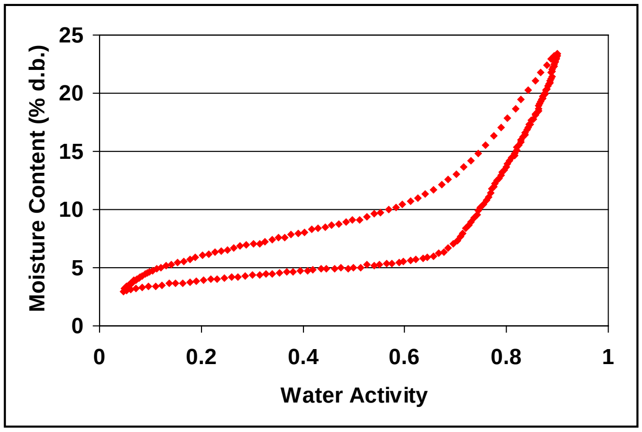

Figure 1. Moisture sorption isotherm for dry pet food generated using the AquaSorp Isotherm Generator.

Once the isotherm has been generated, it can be used to indirectly determine moisture content based on a water activity measurement. This is most easily accomplished using a model that characterizes the isotherm. Many different isotherm models have been proposed, but the most commonly used models are the GAB and BET. Decagon has developed another model, called the Double Log Polynomial (DLP) that is superior to the others for modeling complex isotherms. The models are determined empirically using the data collected during isotherm generation and the resulting equation can be used to calculate moisture content using water activity.

Decagon's new AquaLab Series 4, a chilled mirror water activity instrument, has been designed to accept isotherm equations. Using the isotherm equation for a specific product, the Series 4 can determine moisture content from the water activity values it generates. Each product to be analyzed for moisture content will have a unique isotherm model that must be selected using the Series 4 menu commands prior to testing. A Series 4 is required because the test must be conducted at the same temperature as the original isotherm to be valid.

Clearly, the accuracy of this moisture content method relies on the quality of the isotherm and the repeatability of the water activity measurement. To further investigate the feasibility of measuring moisture content by water activity, Decagon Devices investigated the process using several different product types.

Materials and Methods

Nine products were selected for testing that represent a wide variety of types from homogeneous ingredients to complex final products. The products included: milk powder, flour, dry dog food, chocolate syrup, granola bar, potato flakes, solid dosage tablets, whole wheat, and beef jerky. To identify the isotherm curve most appropriate for predicting moisture content, both full and working isotherms were obtained on the each product in duplicate using the AquaSorp Isotherm Generator. The "as is" moisture content in triplicate was determined for all of the products using convection oven loss on drying. Time and temperature settings for loss on drying were based on AOAC recommendations when available. All moisture measurements are expressed as percent dry basis.

To create samples varying in moisture content, 10 sub-samples were taken for each product, 5 of which were wetted by exposure to 100% relative humidity in a sealed desiccator while the other 5 were dried by exposure to desiccated air inside another sealed desiccator. Sub-samples were removed from the desiccators at different times to create samples varying in moisture content. As the sub-samples were removed, they were sealed in jars and set aside until all sub-samples had been removed from the desiccator. The time in the wet and dry desiccators for the sub-samples of each product was adjusted based on the diffusion properties of the product. All sub-samples for a product were then analyzed in triplicate for moisture content and water activity. Moisture content was measured as before and water activity was measured using Decagon's AquaLab Series 3TE.

The isotherm testing results were characterized using GAB, BET, DLP, and linear models. Adsorption, desorption, and working curves of duplicate isotherms were each analyzed separately. Also, shortened intervals better representing the natural moisture content variation range of the product were analyzed for each isotherm curve. Moisture content predicted using average water activity values was compared to average moisture content from oven loss on drying. Standard Error of Prediction (SEP), which is interpreted as the 95 confidence interval for the predicted value around the actual value (smaller value is better), and R^2 value (closer to 1 is better) were used for comparisons between different isotherm curve/model combinations. The relative strength of a secondary method is measured by how well it matches the reference method. For this study, the SEP value can be considered a measure of the ability of the moisture content by water activity method to correctly match reference data.

Since there is no standard for measuring moisture content, a true accuracy cannot be calculated (Isengard, 2001). Accuracy and precision are used interchangeably in moisture content literature, but in reality, only a precision can be determined. Consequently, the best way to compare moisture content methods is by comparing their repeatability. The precision of the oven loss on drying and moisture content by water activity methods was calculated as the average standard deviation of triplicate analyses across all samples for a given product.

Results and Discussion

Moisture content values calculated from water activity agreed well with oven loss-on-drying values for all products as evidenced by the low SEP and high R^2 values (Table 1). The worst SEP value occurred when moisture content was predicted by water activity for beef jerky (1.48%) and the best was for tablets (0.16%). Most secondary methods for moisture content consider an SEP of 0.60% or lower to be acceptable and all SEP values except for chocolate syrup and jerky were close to that range indicating that moisture content by water activity can be considered a viable secondary method for lower moisture products. It also may be possible to develop better predictions with larger data sets for the high moisture products chocolate syrup and jerky. Figure 2 illustrates the excellent level of agreement between the moisture content values predicted from water activity and the moisture contents determined using oven loss on drying. The unusually low R^2 value for tablets resulted from the small variation in moisture content across samples (very flat isotherm). Table 1 indicates that no single isotherm/model combination was preferred for all product types.

Table 1. Isotherm curve and model combinations that provided the lowest SEP values for each product type.

Product	Isotherm	Model	Rep	*SEP	R^2
Milk Powder	Working	Linear	1	0.58	0.97
Flour	Working	DLP	1	0.52	0.99
Pet Food	Working	GAB	2	0.29	0.99
Choc. Syrup	Adsorption	GAB	2	0..89	1.00
Granola	Adsorption	GAB	2	0.67	1.00
Potato Flakes	Adsorption Short Interval	Linear	2	0.64	0.97
Tablets	Working Short Interval	DLP	2	0.16	0.45

Wheat	Adsorption	GAB	2	0.29	1.00
Jerky	Short Interval Adsorption	DLP	1	1.48	0.96

* SEP values are in % moisture (d.b.)

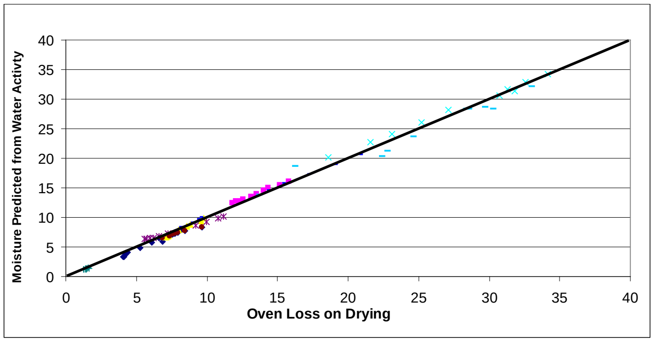

Figure 2. A comparison of moisture contents predicted by water activity (y-axis) to moisture contents determined using oven loss on drying (x-axis) for milk powder (♦), flour (■), dry pet food (▲), chocolate syrup (X), granola bar (*), potato flakes (●), tablets (+), wheat (-), and beef jerky (--). The solid line represents the 1:1 complete agreement line.

Table 2 shows a comparison between the precision of the oven loss on drying method and the moisture content by water activity. For every product investigated, moisture content by water activity gave better precision, even though loss on drying is considered the reference method. Table 3 shows that in comparison to reported precision values for other methods, moisture content by water activity has the highest level of precision.

Table 2. Average precision values for oven loss on drying and moisture content by water activity for all of the products analyzed. The values represent an average of standard deviations of triplicate moisture analyses across 10 samples for each product.

Product	Oven LOD Precision (% Moisture d.b)	Moisture by a_w Precision (% Moisture d.b)
Milk Powder	0.217	0.023
Flour	0.09	0.013
Pet Food	0.121	0.004
Choc. Syrup	0.688	0.077
Granola	0.122	0.01
Potato Flakes	0.170	0.003
Tablets	0.187	0.001
Wheat	0.217	0.019
Jerky	1.068	0.118

Table 3. Commonly reported precision (also reported as accuracy) values for most frequently used moisture content determination methods.

Method	Precision (Accuracy) (% Moisture)
Moisture content by Water Activity	0.001-0.118
Drying Oven	0.1-0.5
Infrared Drying	0.1-0.5
Halogen Drying	0.1-0.5
Microwave Drying	0.1-0.5
Distillation	1
Karl Fischer	0.05-0.5
Infrared Spec	0.3-1
Microwave Spec	0.3-1
NMR Spec	0.1
Gas Chromotagraphy	0.01-0.1

Conclusion

Moisture content by water activity is an excellent moisture content measuring option and is especially attractive when both water content and water activity measurements are needed on the same sample. A product specific isotherm is needed, which can be obtained manually or using an isotherm generator. The precision of this method is the best of any of the secondary methods, and exceeds that for loss on drying. The accuracy can not be assessed because there is, to date, no absolute method for measuring moisture content.

Reference List

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