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The Art of Precision Measurement

Manual

Moisture Sorption Profiles

Step by Step Instructions

LABMASTER NEO

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Dok. Nr.:006777.01

Supplies

1. LabMaster NEO water activity meter -
2. NEO Sorption Isotherm Kit -
3. Salt Slurry Standards at 6 levels (included with the purchase of the instrument)
4. Acquire additional salt standards if needed
5. Sample cups with lids (included with the purchase of the instrument)
6. Isotherm Spreadsheet Excel file
7. High resolution scale with shroud capable of resolving to 0.1 mg or better

The Moisture Sorption Profile

Water profoundly influences product attributes such as quality and safety. To completely understand water relations 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 vapor 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.

Moisture sorption isotherms can be very valuable information to a company. For anyone who dries or wets their product, the sorption isotherm serves as a drying and wetting curve and provides information about the moisture content of a product when dried or wetted to a specific water activity. For anyone who still relies on a moisture content specification instead of a water activity specification, the sorption isotherm will clearly illustrate the potential problems that can occur when you wet to a moisture content vs. dry to a moisture content. The moisture content associated with a safe water activity on the drying curve will not be associated with the same water activity on the wetting curve due to hysteresis. If the production technician controlling the drying of a product over dries beyond the specified moisture content and decides to re-wet the product back to the moisture content specification, the results could be disastrous. The products response to moisture is no longer following the drying curve but has switched to the wetting curve. The moisture content specification may correspond to a safe water activity on the drying curve, but on the wetting curve, the moisture content may be related to a very unsafe water activity level.

An additional function of the isotherm is moisture content prediction. Although water activity is a much better predictor of safety and quality than moisture content, there are times when it is necessary to know both water activity and moisture content as well as the relationship between the two parameters for a given product. Water content measurements can be inaccurate, time-consuming and require a precision balance. Thankfully, the sorption isotherm can be used to predict moisture content based on water activity, usually with better accuracy than actually running a moisture content analysis and in much less time. The prediction is possible

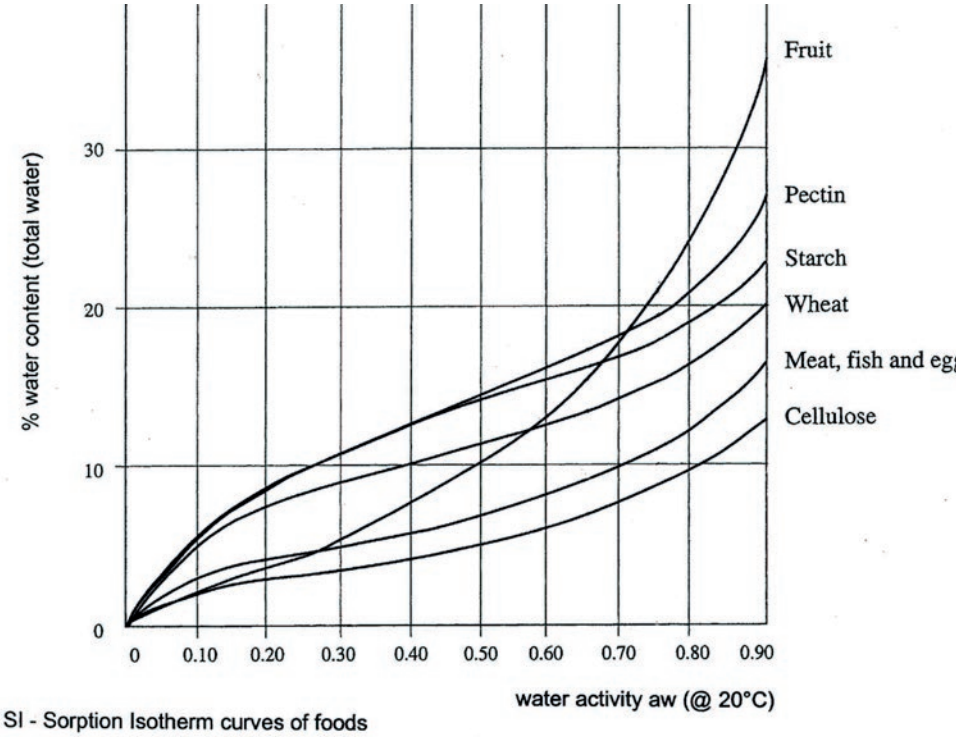
through the use of moisture sorption isotherm models that provide equations that characterize the shape of the curve and give the moisture content at a given water activity. The most common model that provides the greatest level of utlility is the GAB equation:

$$m = \frac{m_o k_b c\, a_w}{(1 - k_b c)(1 - k_b a_w + k_b c\, a_w)}$$

Where m is the moisture in g/100 solids or g/g solids, kb is a constant in the range of 0.70 to 1 and c is a constant in the range of 1 to 2000. In addition, mo is the monolayer moisture content in the same units as m and aw is the water activity at moisture m.

Isotherms can also be very valuable for formulation and product development. By comparing the isotherms of different formulations, it is possible to determine if a product can be adjusted to allow higher moisture content at a given water activity or a lower water activity at a given moisture content. The result can be a moister product that is still shelf stable. For those producing multi-component products, it is possible using the isotherms of the two components to determine what the final water activity will be of the mixture without actually making the product. For dried products, the isotherm will predict the moisture content of the product when it is dried to a shelf stable water activity level.

Finally, sorption isotherms are valuable for shelf life prediction. A product's isotherm can be used to determine package requirements depending on the products sensitivity to moisture. The shape of the isotherm curve can provide insights into the critical water activity that leads to glass transition and subsequent caking, clumping, and loss of texture. Temperature can also impact shelf life and sorption isotherms for a product conducted at different temperatures make it is possible to determine the impact of temperature abuse on moisture content and water activity.



SI - Sorption Isotherm curves of foods
(also called water vapour isotherms)

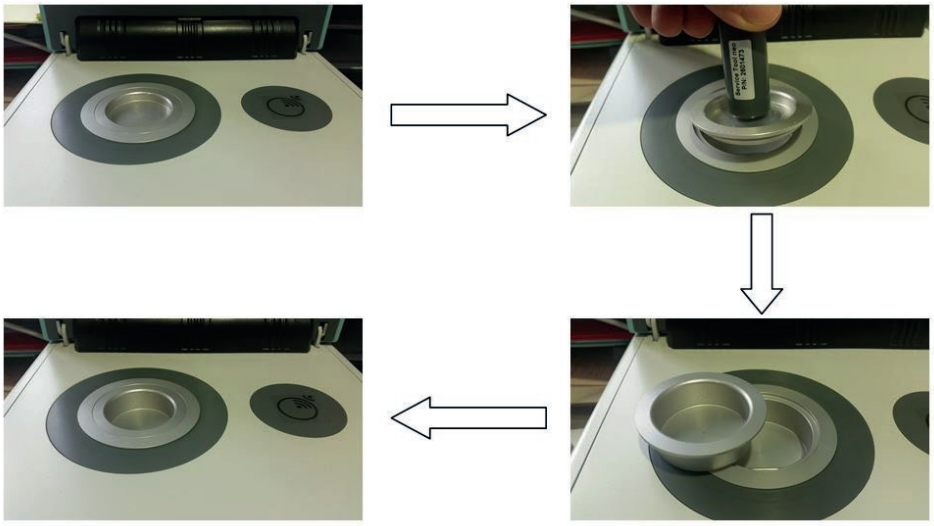
Moisture Sorption Isotherm Determination

1. Adjust the set temperature of the NEO to the desired isotherm temperature
2. Prepare the sample
   a. Obtain a sample of the product of interest and sub-divide into 3 sub-samples.
   b. Use 2 sub-samples to obtain an initial moisture content using a standard method in duplicate and record in isotherm excel spreadsheet
   c. Use the other sub-sample to create the sample for isotherm testing
      I. If a working isotherm is desired, the sample can be used as is, but the sub-sample must be divided again, with one sample used for adsorption at water activities higher than the as is water activity. The other divided sample will then be used for water activity levels below the as is water activity. (See section 9 for more information about conducting working isotherms)
      II. If an adsorption curve is desired, the sample will be placed in the NEO with the lowest Salt-T standard first, following the instructions in Step 4 to setup the test. Then, additional SAL-T of increasing water activity will be used sequentially.
      III. If a desorption curve is desired, the sample will be placed in the NEO with the highest Salt-T standard first, following the instructions in Step 4 to setup the test. Then, additional SAL-T of decreasing water activity will be used sequentially.
3. All measurement values will be recorded in the Data Collection Sheet of the Isotherm Spreadsheet Excel file. Rename the file to reflect the current test so as to preserve the Isotherm Spreadsheet file as a template.
   a. Enter the initial moisture content of the sample in the Initial Moisture Content Excel cell at the top of the worksheet.
   b. Enter the as-is water activity of the sample in the Initial Water Activity Excel cell at the top.
   c. Weigh and record the weight of the glass weighing container with lid and the small metal sample cup with mesh bottom and enter it into the Excel cell at the top labeled Empty Dish and Lid Wt. This will be the tare weight for all samples. The small metal cup is most easily handled using the included tongs.

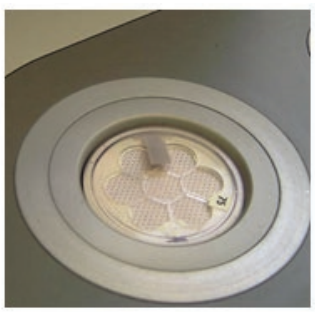
d. Do not enter anything in the Initial Sample Weight cell as this will be updated automatically later.

4. Begin the test
   a. Enter the water activity value of the current salt standard being used in the water activity column in the Excel cell corresponding with the current test number. The water activity will change for each test number according to the pre-selected salt standards that will be used to create the isotherm.
   b. Place the isotherm sample into the small metal sample cup, filling it half to three quarters full.
   c. Place the small metal cup with the sample in the glass weighing container with the lid on. Zero the balance then weigh everything (sample cup + sample + glass container + lid) and record in the Data Collection sheet in the Start Weight column corresponding with test number 1. The Initial Sample Weight in the Excel cell at the top will automatically adjust to show the actual initial weight.
   d. Record the current time in the Start Time column in the Excel cell corresponding with the current test number.
   e. The easiest way to enter the current time is to type '=now()' in a cell and hit enter. The current date and time will now appear. To prevent the time from constantly updating, copy the cell and choose to paste special: Values & Number Formatting in the desired cell, this will lock in the weigh time.

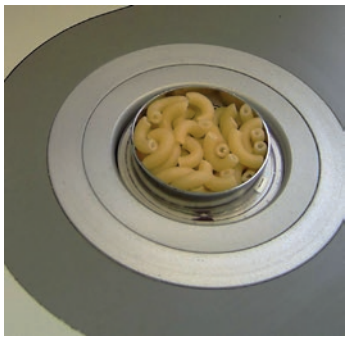
5. Prepare NEO



   a. Open the NEO and remove the chamber cup using the Multi Tool included with the NEO



and replace with the chamber provided in the SI Kit.



b. Add the desired salt standard to the SI chamber
c. Place the small metal cup with the sample, into the NEO Chamber on top of the salt std.
d. Close the lid but do not start a water activity test.

6. Sample Equilibration
   a. Watch the water activity reported on screen, which will update in real time, until it matches the reported water activity of the standard. This may require several hours or up to a day.
   b. When the water activity of the screen is within +/-0.003 of the reported water activity of the salt standard, record the water activity reading on the instrument screen in the On-Screen Aw column in the Excel cell corresponding with the current test number
   c. Open the lid of the instrument and remove the small metal sample cup and place it in the glass container with its lid.
   d. Weigh everything (sample cup + sample + glass container + lid) record it in the excel sheet as End Weight.
   e. Record the current time (using the =now() function as explained earlier) in the End Time column in the Excel cell corresponding with the current test number. The Time Elapsed column will automatically be updated.
   f. The moisture sorption isotherm curve at the bottom will automatically update as the final weight is recorded.
7. Repeat Steps 5-7 for remaining test numbers and corresponding desired salt standards.
8. Test Finalization: Transcribe the water activity and %moisture content results into the Isotherm Modeling sheet to see a graph of the data and do GAB modeling.
9. Special Note about Working Isotherms
   a. If a working isotherm (adsorb and desorb from as is water activity) is desired, the test must be done twice, once for water activities lower than the as is and once for water activities higher than the as is for the sample.
   b. 2 separate samples and 2 different Data collection sheets should be used for the desorption and adsorption curves, but the initial water activity and moisture content will be the same for both. The Initial Sample Weight will however be unique to each sample for each desorption and adsorption curve.
   c. The data can be combined into one isotherm by transcribing the final results for the desorption and adsorption curves into the Isotherm Modeling worksheet.

Moisture Sorption Isotherm Modeling

1. Copy the water activity and moisture content data from J26 to K37 in the Data Collection excel sheet and paste it in cells B8 to C19 on the Isotherm Modeling excel sheet in the Isotherm Excel Spreadsheet. Past the values over any existing data and remove any extra data points. Copy and paste from the existing GAB and Sq Diff cells into any additional cells needed to match the water activity and moisture content data.
2. In excel, choose the Data menu at top and then choose the Solver function. You may need to add this function if it does not show up in the menu. To add, refer to excel help section.
3. In solver window that opens, for the “Set Objective” cell, choose the cell next to SSE (E19 in template).
4. Then for the next line labeled To:, click on the circle next to Min
5. Finally, for the “By Changing Variable Cells:”, select cells H5 to H7. Should now show $H$5:$H$7 in the space.
6. Click on the Solve button at the bottom of the window and the computer will change the values for Monolayer (mo), C1, and k until it finds the values that minimizes the sum of square differences between the actual moisture contents in column C and the GAB values in Column D.
7. Chose OK in the Solver Results window that appears to accept the results.
8. Verify that the R2 value below the constants is above 0.90, with a value closer to 1 showing better agreement between the GAB model and the actual data.
9. The values that now appear in cells H5-H7 are the constant values for the GAB Isotherm Equation as shown in the center of the sheet.
10. The figure graph labeled Moisture Sorption Isotherm
11. The cells shown under “Moisture Content Prediction from Water Activity” can now be used to predict the moisture content at any water activity. The cells under Moisture Content use the GAB equation and the derived constants to make this prediction.