# Moisture Sorption Isotherm Report

Company: Addium, Inc.
Contact: April Addium
Address: 2365 NE Hopkins Ct.
Pullman, WA 99163
SO: SO-000000
Scientist: Andy Addium
Technician: Abigail Addium
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## Background

From the request form:

We are interested in determining the transition points for two products at two temperatures.

## Materials

| Sample | Identification | Description |
|---|---|---|
| 1 | Sample 1 | Powder – Lot LKJSDI465 |
| 2 | Sample 2 | Powder – Lot X4LSK352 |

*Table 1 – Sample identification and descriptions for all samples tested.*

## DDI Isotherm Settings

| | **Test 1** | | |
|---|---|---|---|
| **Samples** | 1, 2 | | |
| Sorption | Adsorption & Desorption | | |
| Sensor | Dew Point | | |
| Temperature | 25 °C | | |
| Flow Rate | 80 mL/min | | |
| $a_w$ Range | 0.05 – 0.50 | | |
| Resolution ($a_w$)[1] | 0.01 | | |
| Timeout[2] | Off | | |

*Table 2 – Dynamic Dewpoint Isotherm (DDI) settings used for testing in the Vapor Sorption Analyzer (VSA).*

## Sample Handling and Preparation

Two samples arrived in screw top plastic jars inside zip top plastic bags and were stored at room temperature until the time of testing. Immediately prior to testing the package was opened and approximately 1 g was transferred to a stainless steel sample cup. It was placed directly into the VSA and the test started to limit exposure to ambient conditions. Additional sample was taken for water activity and moisture content testing.

[1] The resolution for DDI determines the target $a_w$ resolution. A higher resolution value makes the test faster, but decreases the $a_w$ resolution.
[2] Maximum amount of time allotted to each sorption.

# Results & Interpretations

Raw data is available in a separate Excel workbook. VSA files available upon request.

Both samples submitted for analysis exhibited a slope change indicative of a phase change in the material as it takes up water (Figure 1). This phase change is indicative of a glass transition event as the material changes from a glassy state to a rubbery state. Since this transition will end shelf life, this water activity is referred to as the critical water activity ($RH_c$).

Prior to the transition, the material shows substantial changes in water activity but very little change in moisture. This lower water activity range comprises primarily surface adsorption. As water activity is increased past the transition point, the material undergoes a physical change and begins to take on more water as additional water binding sites become available.

In Figure 1, each sorption is shown separately with all together for comparison in Figure 2.

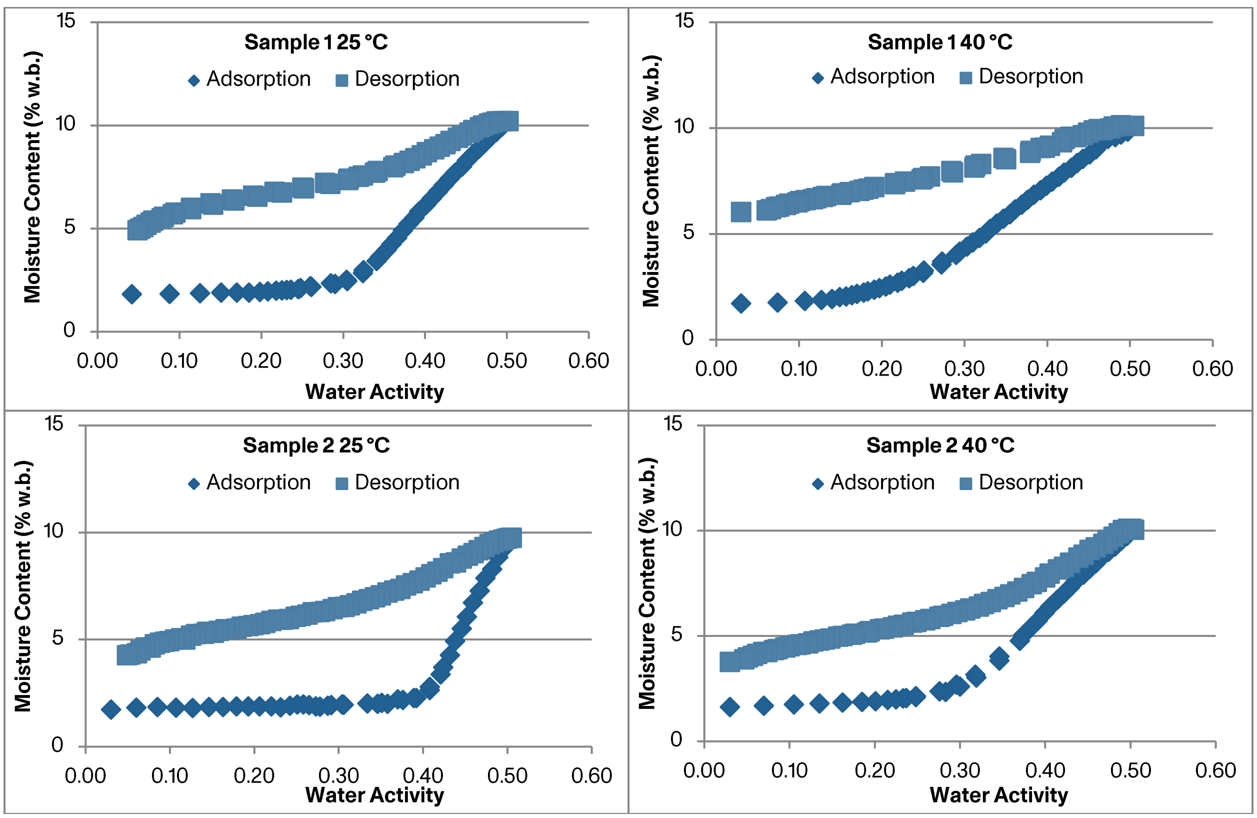


*Figure 1 – Full moisture sorption isotherms for each sample and temperature tested.*

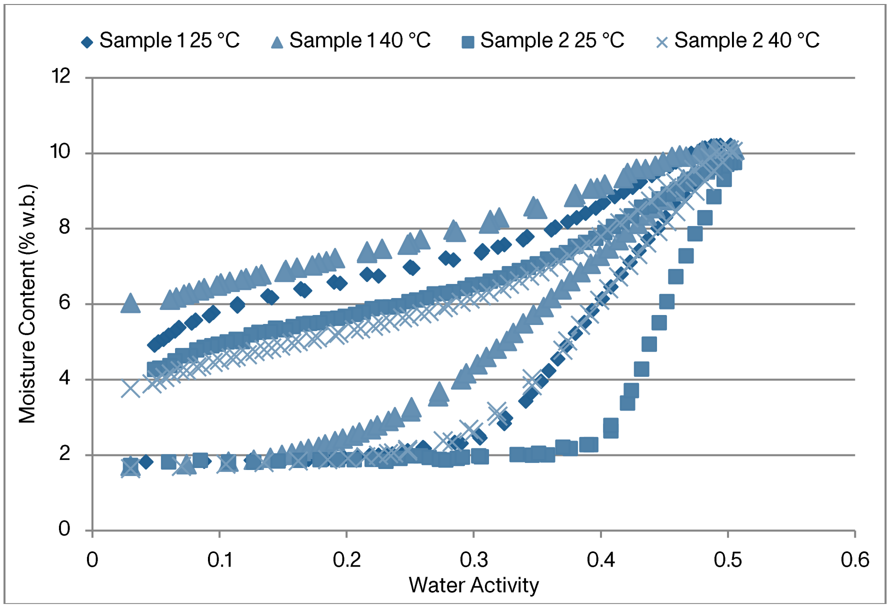


*Figure 2 – All moisture sorption isotherm tests overlaid.*

Figure 3 shows the Savitzky-Golay second derivative analysis for each adsorption curve. The second derivative is useful in identifying slope changes in the original curve. Slope changes in the adsorption curve are the point at which there is a physical change. The peaks correspond to any significant change in slope of the curve. As discussed above, the transition is a clumping point, the $RH_c$.

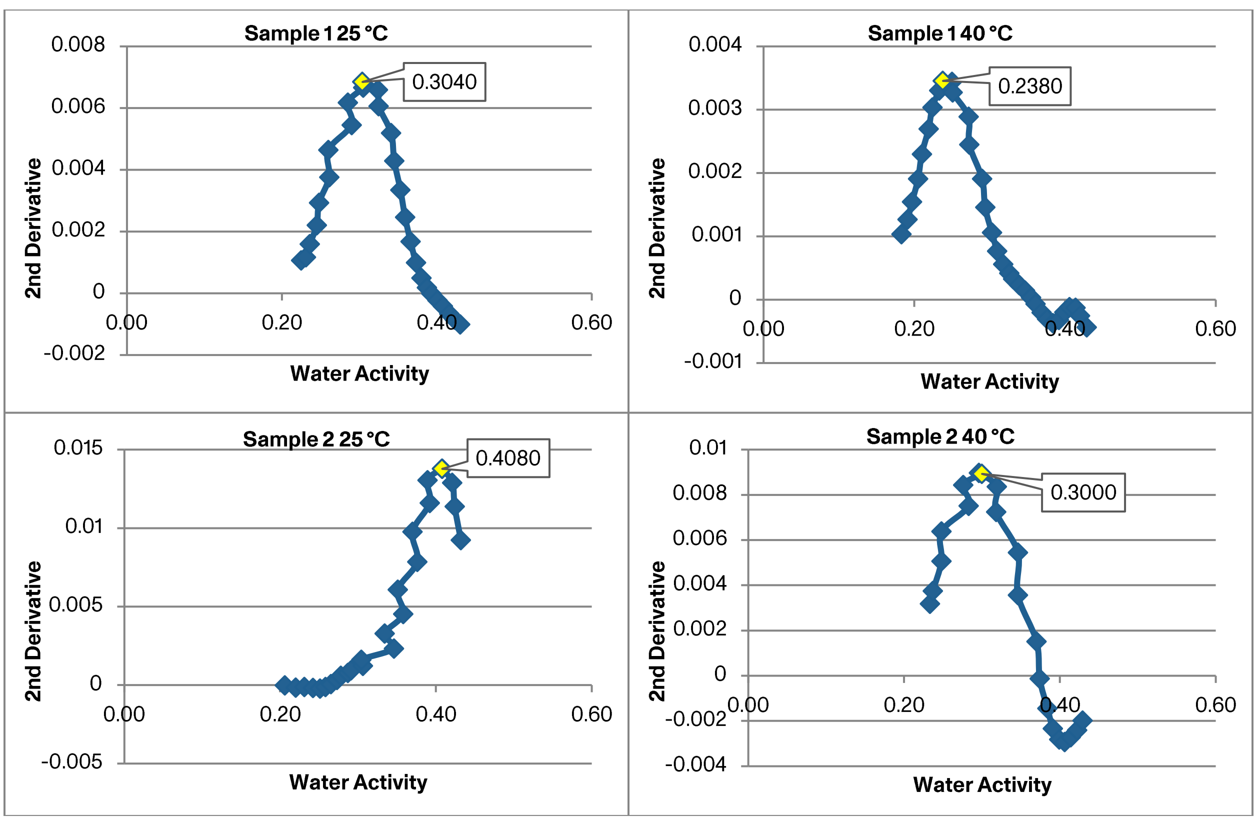


*Figure 3 – Savitzky-Golay second derivative analysis of the adsorption curves seen in Figure 1.*

Addium, Inc.
1300 NE Henley Court, Pullman, WA 99163
**T** 509.332.6000
**E** support@aqualab.com **W** aqualab.com

| Sample | Temperature (°C) | Transition Point $a_w$ |
|---|---|---|
| 1 | 25 | 0.304 |
| 1 | 40 | 0.238 |
| 2 | 25 | 0.408 |
| 2 | 40 | 0.300 |

*Table 3 – Identified transition points for each sample and temperature.*

To maintain the ideal properties of the powder and flowability, the product will need to be maintained at water activity levels below the $RH_c$. If the product increases to the $RH_c$ or above, it will begin to cake and clump. Sample 1 has a lower $RH_c$ value indicating it will transition more readily than Sample 2. Appropriate packaging will be needed to protect the product from ambient humidity in order to maintain the product properties.

This trend continues with the higher temperatures. The slopes calculated in Figure 4 can be used to estimate transition points at other temperatures in the range. The attached Excel workbook contains calculated values on Tab 1.

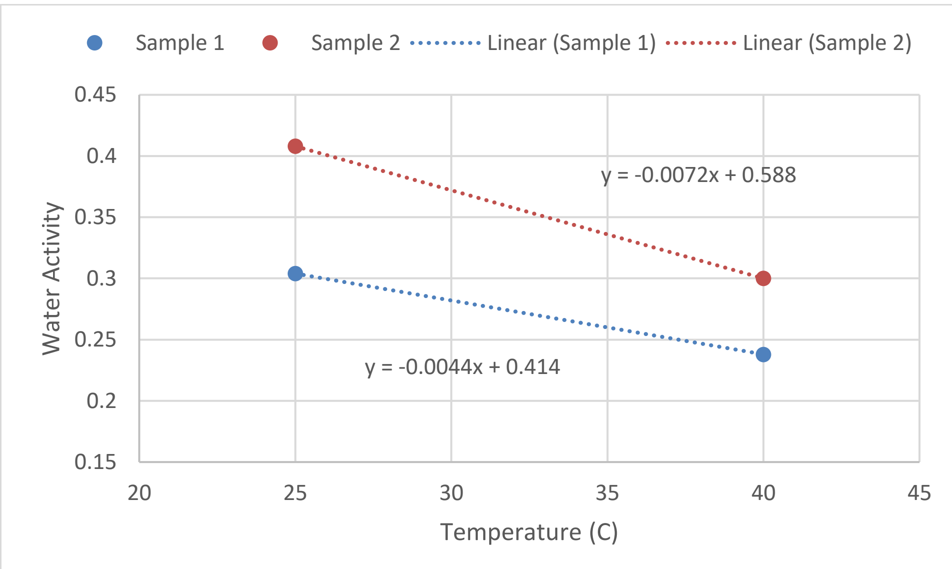


*Figure 4 – Transition points by temperature with trendlines and equations.*

The identified transition points can be used for setting manufacturing specifications, used in subsequent shelf life calculations, and taken into account when setting environmental conditions in the manufacturing facility.

The curves can be used to build models that will predict the moisture content from a water activity reading. AQUALAB water activity readings generally take between 3 and 5 minutes but can take 1 minute if the SKALA software is used. Moisture content readings often take multiple hours for a normal Loss on Dry method.

## Conclusions and Next Steps

- Clumping points were able to be determined for each sample at two temperatures (Table 3).
- The relationship between the clumping points and temperature was determined. Using these equations, transition points can be determined at any other temperature within the normal range above freezing.
- Consider future shelf life analysis to determine appropriate packaging for a desired shelf life for each powder.
- Consider utilizing the AQUALAB SKALA platform to reduce moisture content test times by measuring water activity and utilizing the isotherms measured in this report to calculate the corresponding moisture content.