IAFP’s Avoiding Premature Water Activity Testing Results When Meeting Safety Regulations

Organized by: The Low Water Activity Foods PDG

Moderator: Laure Pujol, Novolyze, France

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Laure Pujol, Novolyze

Laure Pujol is a Food Safety and Quality Expert at Novolyze. She has a Ph.D. in Predictive Microbiology and Risk Assessment from ONIRIS & INRA in Nantes, France, and a Food Engineering Diploma. As a Preventive Control Qualified Individual (PCQI) and a process authority recognized by the Technical Expert Review Panel (TERP) and Almond Board of California (ABC), Laure is very experienced working with low water activity foods and has performed in-plant validation trials around the world. She is an active member of the PDG Low Water Activity Food at IAFP and is part of the ASTA Validation Task Force. She organized symposium at the IAFP EU and participate to several scientific conferences helping food processors managing their food safety and quality issues.
Today’s Panelist

Brad Taylor
Brigham Young University

Dr. Brad Taylor received his doctorate in Nutrition & Food Science at Utah State University (USU). Brad joined the faculty at Brigham Young University (BYU) from the WhiteWave Foods Company (now Danone NA) where he served as the Sr. Director of Research Sciences and Regulatory Affairs. He is an Associate Professor of Food Science in the College of Life Sciences with an even split between teaching and research.

Brad teaches food processing & technology courses including an introduction to food chemistry course, a course on food regulations and graduate courses on carbohydrates in foods and other topics relevant to applied food microbiology and the global food supply. Priority research areas include: 1) applied research in food safety & preservation technologies, 2) characterizing emerging microorganisms of concern and beneficial bacteria in low-moisture food matrices and 3) novel food / ingredient utilization or waste avoidance.
Dr. Brady Carter is a Senior Application Scientist with Neutec Group. He specializes in Water Activity and Moisture Sorption applications. Dr. Carter earned his Ph.D. and M.S Degree in Food Engineering and Crop Science from Washington State University and a B.A. Degree in Botany from Weber State University. Dr. Carter has 20 years of experience in research and development has been the instructor for water activity seminars in over 23 different countries and has provided on-site water activity training for companies around the world. He has authored over 20 white papers on water activity, moisture sorption isotherms, and complete moisture analysis. He has participated in hundreds of extension presentations and has given talks at numerous scientific conferences. He developed the shelf life simplified paradigm and hygrothermal time shelf life model and is the leading expert in applying water activity to shelf life prediction.
Water Activity Test Speed: Sample or Instrument or both?
Why Measure Water Activity?

- Comply with safety regulations
- Release criteria / specification
- Monitor product quality
- Shelf life determination
- Meet or exceed global standards
Water Activity Applications

• Optimizing ingredients and processing
• Prevent moisture migration
• Slow and control chemical reactions
• Optimizing product texture
• Preventing caking and clumping
• Selection of packaging materials
Water Activity Applications (cont.)

WATER ACTIVITY - STABILITY DIAGRAM

- Ionic
- Covalent
- Solute & Capillary

Moisture Sorption Isotherm
- Browning Reactions
- Enzyme Activity
- Mold Growth
- Yeast
- Bacteria

Relative Reaction Rate

Moisture Content vs. Water Activity
Safety Regulations and Methods

Water activity is a critical parameter!

FDA: Definition for Potentially Hazardous Foods
FDA: Food Safety Modernization Act – Risk Based Approach (HARPC)
USDA: HACCP – critical control point
EU PAFF – Included in the Codex of Food Hygiene
UN Food and Ag Organization – Water activity for food preservation
ASTM D8197-18 – Defines acceptable water activity range for Cannabis
Pharma: USP Method <1112> <51>
ISO29621: Microbial Control for Cosmetics
Safety Regulations: Monitoring & Auditing

• While government guidance rarely certifies specific monitoring methods, manufacturers must be able to verify the validity of their monitoring testing results

• Validity of testing results enhanced when identified and spelled out in the food safety program... “testing will be done according to __________, a standard method”

• Instrumentation should be checked, recorded, and calibrated according to the manufacturer instructions using reference standards
Standard Methods

AOAC 978.18
ISO18787
ASTM D8196-18
USP 922

Compendium of Methods for the Microbiological Examination of Foods 5th ed.
Water Activity Testing

... Common Questions

• What is water activity?
• How is it measured?
• What is the best instrument?
• How long does it take?
Theoretical Background

\[ \mu = \mu_o + RT \ln \left( \frac{f}{f_o} \right) \]

- Chemical Potential
- Gas Constant
- Temperature
- Fugacity

Chemical Potential of the pure substance
Correct definition of water activity

*Old Definition:* Water activity is the amount of “free” or “available” water in a product (as opposed to “bound”)

*CORRECT “enhanced” Definition:* Water activity is a measure of the energy status of the water… in a system (at a specific and constant temperature & pressure)

\[ a_w = \frac{f}{f_o} = \frac{p}{p_o} \]

Vapor pressure of water above sample at ___ °C

Vapor pressure of pure water at same °C
Vapor Equilibrium

\[ a_w = \frac{p}{p_o} \]

1. Equilibrium
2. Constant Temperature & Pressure
Test Time Facts

• Water activity is not a 5 minute or less test
• Cannot be measured in 1 minute without a prediction
• All instruments rely on vapor equilibrium
• The test time will vary by product
• An instrument cannot speed up the equilibration process
Test Time

Product 1

Product 2
Determining End of Test

• Vapor equilibrium detected by a slowing of the rate of water activity change to a steady state
• What target rate of change is used and how it is determined will impact test time and accuracy
• Achieving the targeted endpoint only once vs. continuously over a time period is more easily done, but can result in premature results
• Fast test results doesn’t mean good test results if vapor equilibrium has not been achieved
• ISO 18787 specifies the end of test requirements to eliminate ambiguity.
  • 0.0003 $a_w$ difference for 3 measurements or 1 minute
• Reported test times will not likely reflect performance when using this ISO standard
Determining End of Test

The ideal system will allow the user to adjust the end-of-test requirements

• Slow - Most stringent – No difference >0.001 for 6 minutes
• Average – Less stringent - No difference >0.001 for 4 minutes
• Fast – Least stringent - No difference >0.001 for 2 minutes
• Manual – Set the stability time – setting to 3 means to run until no difference > 0.001 for 3 minutes
• Quick Mode – Test ends at 10 min – useful for intermediate checks
• ISO18787 Mode – Uses ISO 18787 specified end of test requirements
Accuracy and Precision

• Accuracy
  • How well the measured value matches with the actual value
  • Requires independent standards of known value

• Repeatability
  • The variability in readings on sub-samples
  • Will vary for different matrices

• Reproducibility
  • The variability in readings on sub-samples taken by different users or on different days

• Precision = Repeatability + Reproducibility
The Importance of Vapor Equilibrium

- Premature water activity = 0.84
- False pass due to not waiting for vapor equilibrium
- End of test only requires meeting a water activity difference one time, so test ends prematurely
- Actual water activity = 0.87
- Fails as it does not meet water activity required levels
Crème Filling Equilibration

![Graph showing the equilibration of Crème Filling with Water Activity and Time. The graph indicates two modes: Fast Stability and Slow Stability.](image)
Gummies Equilibration

![Gummies Equilibration Graph]

- **Gummies**: Dewpoint Continuous Mode
- **Water Activity**: 0.635 to 0.66
- **Time**: 0 to 100
- **Stability**:
  - Fast Stability
  - Slow Stability
Beef Jerky Equilibration

![Graph showing the water activity over time for Beef Jerky, with points indicating Fast Stability and Slow Stability.](image-url)
Equilibration Screen
Vapor Equilibration Summary

• Typically, a 0.02 $a_w$ change between the first and last dewpoint measurement using the default stability settings

• The fast stability setting gives results slower than the first dewpoint data point, but the result is further along the equilibration curve

• The slow stability setting matches the results achieved at the end of continuous testing

• Fast and accurate are usually mutually exclusive
Summary

• Governmental food safety programs place the burden on the manufacturer to produce safe product

• Monitoring tools like water activity assure that preventative controls or begin appropriately applied

• Sacrificing true vapor equilibrium testing results for faster results may result in releasing unsafe product and recalls

• Faster test results may be justified if stringent settings only result in small changes (<0.005 aw)

• However, should always check the impact of fast results – Need an equilibrium curve on the instrument
Contact Information

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May 24  Achieving Best-In-Class Food Safety Culture Through Evolution
May 26  Making Your Environmental Monitoring Plan Smarter
June 23  7-Steps of Sanitation (Spanish)

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