Drying Laboratory Methods – Drying Parameter Characterization from Small Size Samples

By Steve Zagar and Jeff Quass
Why would you care to “watch” drying?

• Drying of new materials in industrial processes can present challenges in “unfamiliar territory”
• What will it take to dry the product without defect?
• Developing a familiarity with drying variables and system responses reduces mystery, potentially leading to...
• Better success in productivity and profitability in coating and drying operations

Developing a familiarity
Drying Experiments → Analysis → Characterization → Process Decisions
How do you “watch” drying?

• Drying development operations can be carried out in:
  ➢ pilot coater lines
  ➢ production facilities
  ➢ laboratory settings

**Caution:** Drying results obtained in one of these settings may not necessarily (but can) translate well to another depending on process similarity, measurement gages and methods used.
How would you “watch” drying?

So, how do I develop a familiarity?

Proposition: You endeavor to make enough observations of the process and become more familiar...

That’s it, I’ll just fire up the trusty old pilot line.
Typical venue to watch drying

• Drying development operations in pilot coater lines
  ➢ Depends on availability of the appropriate facilities
  ➢ Requires moderate amounts of material – typically gallons of coating and hundreds if not thousands of square feet of web substrate
  ➢ Are intended to simulate actual process conditions with moving web
  ➢ Generates valuable process data for scale-up and troubleshooting information, depending on capabilities of instrumentation

The output of the pilot line is data... and some sample product too.
Alternate venue to watch drying

• Drying development operations in production facilities
  ➢ Depends on availability of the appropriate facilities, displacing production time slots for saleable product
  ➢ Requires larger amounts of material, most or all of which will typically become scrap
  ➢ May produce limited process data for taking production elsewhere, depending upon instrumentation and sensors
  ➢ As they are run under full-scale production conditions, results represent the real performance – on this line

  *If I can make it here, I can, well ... make it here!*
A smaller venue to watch drying

So, how do I become familiar if...

• I do not have an existing process?
• I have barely a beaker of coating and maybe a few patches of substrate
• I need some data now to make process design decisions

Revised proposition: You can use bench-top hardware and make careful observations on small specimens

It’s not even moving!
What about laboratory methods?

• Drying development operations in laboratory settings can be:
  ➢ Crude but useful, as in a simple dry down batch oven, or
  ➢ Sophisticated as in a highly instrumented test chamber
  ➢ A useful step in early scale-up work or process troubleshooting
  ➢ The only practical venue available when test materials, time or other resources are scarce

*Never in the field of web drying was so much learned from such a small oven with so little sample...*
Drying Laboratory Methods

Some Prior Examples and References for Small Sample Bench Top Work

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Small Sample Drying Apparatus

Present Work: Flame Ionization Detector, Humidity & Temperature Sensors allow measurement of aqueous and organic solvents including combinations thereof

Bench Top Drying Apparatus
Small Sample Drying Apparatus
Overview of Drying Oven Method

• A wet coated sample up to 12 cm x 12 cm is placed in the preheated sample chamber and supported as needed on a wire frame
• Bone dry air (typically less than -50°C dew point) is supplied to the drying chamber and directed by jet nozzles to sweep over one or both sides of the specimen to obtain the desired heat and mass transfer coefficients
• Oven supply air temperature is set and held at the desired level in the apparatus with a set of electric heaters and controllers
• Moisture content in the air exhausting from the drying cell is measured by a humidity sensor having high sensitivity (± 1.5% RH resolution) and fast transient response (~10 seconds or less)
• Solvent concentration in the air exhausting from the drying cell is measured by a flame ionization detector with resolution down to ~1 ppm C3 by volume
• Numerical integration of the data logged humidity and solvent concentration curves produce a drying history
• Drying measurement of coating moisture and solvent levels down to less than 1% residual exhibit good resolution and repeatability
Small Sample Drying Laboratory Results

What types of info can I hope to get?

• For the process formulator
  ➢ Estimate of drying time and temperature requirements for coatings
  ➢ Qualitative drying behavior of coatings – blistering, binder migration, etc.
  ➢ Comparison of formulation options

• For the plant process engineer
  ➢ Falling rate critical moisture, zone temperature profiles, drying times
  ➢ Target humidity and/or solvent LFL levels
  ➢ Drying profile settings for avoidance of defects

• For the drying process engineer
  ➢ Drying data for dryer hardware sizing (drying model inputs)
  ➢ Drying oven process strategy – zones, temperatures, velocities
  ➢ Falling rate period(s), critical moisture, temps
  ➢ External and internal mass transfer rates and coefficients, (k/h, internal diffusion coefficients)
Drying Curve from Laboratory Apparatus

Example 1 – Nonwoven Specimen/ Aqueous & Isopropyl Alcohol Solvent

Drying Lab 3 Drying Curve

**Sample:** Nonwoven on PET Carrier/ Aqueous & IPA

- **Water lb/lb dry basis**
- **Solvent lb/lb dry basis**
- **Specimen Surface Temp °F/100**
- **water rate lb/hr ft²**
- **solvent rate lb/hr ft²**

**200 °F Test Cell Temperature, Flow = 10 SCFM**

- **Drying Rate Peaks for IPA – No Constant Rate Observed**
- **Constant Rate Period for Water**
- **Falling Rate for Water Begins**
Extended View of Drying Curve

Example 1 – Nonwoven Specimen at Low Residual Moisture & Solvent

Internal Diffusion Coefficients may be Determined from Slopes
Solvent Drying Curve from Laboratory Apparatus

Example 2 – NMP-based battery anode slurry

No Constant Rate Period Observed

Internal Diffusion Coefficient may be Determined from Slope
Drying Laboratory Methods

What else can we see?
• Add a video camera to look at the specimen during drying

Field of View: 1.3 cm x 1 cm
Drying Curve from Laboratory Apparatus

Example 3 – Water-based battery anode slurry

Temperature inflection time corresponds to onset of mud cracking
Notable Findings to Date

• Laboratory methods for drying small sample coupons for the purpose of process characterization and scale-up have advanced over the past several decades

• Insights gained by these methods have proven valuable in dryer selection and design over the years

• Qualitative, semi-quantitative and even precise drying measurements suitable for early research work are possible with the proposed laboratory methods
Future Work and Applications

• Methods presented offer a number of “windows on the process” providing enhanced capability in formulation development work while requiring relatively small sample amounts to conduct experiments.

• Design and selection of candidate drying methods can be made early in the development of new coatings to help assess commercial viability of scale-up to production.

• Analyses and troubleshooting of drying issues on existing processes are also possible with the benefit of conducting work off-line and with reduced material requirements.
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Thank You!

Questions?

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