ON THE QUALITY LINE

PERMEABILITY TESTING:
THE CHALLENGES AND SOLUTIONS

PBI Dansensor
Lyssy Line of Permeability Testers
Agenda of Presentation

• Permeability Theory
  – Mechanism of Permeation
  – Factors Affecting Permeation Rates
  – Challenges of Permeability Measurement

• Water Vapour Permeation
  – Traditional measurement method
  – Automatic measurement method

• Oxygen Permeation
  – A new automatic measurement method
Model of Permeability Phenomenon

Gas or vapour transfer through a package wall

Phase 1 Phase 2 Package Wall

Sorption

Desorption

Diffusion (Fick’s Law)
Variables Affecting Permeation & Diffusion

1. Compositional Variables:
   a) Chemical composition of polymer and penetrant
   b) Morphology of the polymer
   c) Concentration of the penetrant
   d) Presence of copermeant

2. Environmental and Geometric Factors
   a) Temperature
   b) Relative humidity
   c) Packaging geometry
Typical Permeation & Time Lag Curve

\[ Q \text{ (ml at STP)} \]

\[ L \]

\( Q \): amount of penetrant permeated as a function of time \( t \)

\( L \): time lag
Challenges of Permeability Measurement

- Precise control of key parameters essential
  - Testing temperature
  - Permeant driving force
  - Relative humidity

- Measurement can be time-consuming
  - Conditioning time (before testing begins)
  - Repeated tests (to ensure steady state)
  - High barrier materials take even longer to measure
Challenges of Perm. Measurement (2)

• "True" Permeabilities not easily verified
  – No published values for laminates, composites, coatings
  – Variations can exist within a single polymer
  – Temperature relation is material-dependent

• Interpretation of Results
  – Permeability, permeance, transmission rate
  – Influence of ambient conditions (atm. pressure)
Water Vapour Permeability Testing

• Some typical Applications:
  – Food packaging: uniform and optimised shelf life ensures high-quality product
  – Pharmaceutical packaging: product protection (e.g. Shelf life), maintenance of dry or aqueous conditions
  – Electronics: protection of components
  – Construction: weather-proofing, optimising interior atmosphere
  – Textiles & Hygiene: maximum personal comfort and optimal product function
Gravimetric Permeability Measurement Methods

Cup method Absorption („Dry Cup“)

High RH (climate chamber)

Absorbent material

Cup method Desorption („Wet Cup“)

Low RH (climate chamber)

Water or salt solution
Challenges of Gravimetric Method

• Correct sealing of cup is essential
• Climatised chamber required
• Labour-intensive preparation of samples
• Time-consuming, especially for high barrier materials (up to 4 weeks or more for 1 test)
• Reproducibility is sometimes poor
• Salt solutions messy and corrosive
Automated WVTR Testing: Dynamic Relative Humidity Measurement

**Measuring Principle**: Compare time required for incremental RH increase in Upper Chamber (i.e. 9.5 → 10.5 % RH)
Measuring Cycle of Automatic WVTR Tester

Upper limit

Lower limit

Underdry cycles

Measuring interval

Humidity compensation cycles

Relative humidity

10.1%

10%

9.9%
Advantages of Automatic WVTR Tester

- Humidity sensor directly in test chamber
- Measuring time: inverse relation to WVTR
- Fast results even for high barrier materials
- Measurement of both low and high WVTR
- Sample preparation and changing easy
- Self-contained instrument: no PC needed
- Very little maintenance required
Oxygen Permeability Testing

• Some typical Applications:
  – Food packaging: uniform and optimised shelf life, prevention of product degradation (e.g. microbial growth, oxidative rancidity)
  – Pharmaceutical packaging: product protection and shelf life (prevention of oxidation reactions)
  – Electronics: protection of components
  – Medical products: permeable membranes (e.g. Gas-permeable contact lenses)
Gas Permeability Testing: Differential Pressure Method

Key to Components:
1: Transmission Cell
2: Film Sample
3: Filter Paper
4: Cell volume-control device
5: Pressure Sensor
6: Gas Feeder
7: Vacuum Pump
8: Gas Supply
9: Stop Valves
Limitations of Differential Pressure Method

• Poor sensitivity at very low OTR rates
• Pressure gauges "blind" to gas type being measured
• Generally limited to dry gas: measurements with humidified gases required complicated calculation procedure
Oxygen Permeability Measurement: Equal Pressure Method

F: Flow regulation
H: Humidifier
S: Sensor
OM: Gas outlet manifold
Zirconium Oxide Sensor

Measures Oxygen Partial Pressure
Measuring principle follows Nernst Equation
Reference: Atmospheric Air (20.9% O₂)
Measuring range: 0.01 ppm - 100% O₂
Reproducibility: Better than 10 ppb O₂
Sensor lifetime: 5 years or more
Zirconium Oxide Sensor (2)

- Sensor signal proportional to **Logarithmic O₂ partial pressure**
- Logarithmic signal yields **high** sensitivity at **low** O₂ partial pressures
- Sensor Millivolt output → O₂ concentration
OTR Tester Measuring Principle

- Reference measurement (bypassing the chamber)
- Sample measurement (through the chamber)
- \( \text{O}_2 \) conc. difference is proportional to permeability
Advantages of New Automatic OTR Tester

- Broad range of OTR values (high & low)
- Precise control of test and carrier gas relative humidity
- Long sensor lifetime (5 years or more)
- Easy to operate
- Self-contained instrument: no PC needed
- Very little maintenance required
Summary

• Permeability Testing important to development of high-tech packaging solutions and assurance of product uniformity
• Permeation theory can be complex, and measurement is sometimes time-consuming, labour-intensive and challenging to verify results
• Automated permeability testers provide faster, easier and reliable testing:
  – WVTR: Dynamic Relative Humidity Measurement
  – OTR: Equal Pressure Method with Zirconium Oxide Sensor
Thank you for your Attention!

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