La Spectroscopie Résolue Spatialement (SRS) : Comment mieux mesurer les spectres et caractériser absorption et diffusion

Challenge : VIS-NIR Spectroscopy for complex liquid and solid analysis

Sylvie ROUSSEL, *PhD* - <u>sroussel@indatech.eu</u> Fabien CHAUCHARD, *PhD* – <u>fchauchard@indatech.eu</u>



385 Avenue des Baronnes 34730 Prades le lez France www.indatech.eu

1

- What are "Complex products"
- Principle of Spatially Resolved Spectroscopy (SRS)
- Light/Matter interaction simulations
- Chemometrics
- INDATECH solutions
- Application examples



Complex Solid Products

- Highly-scattering solid media :
 - Powders, Granules, Particles compressed into a matrix, multilayered materials
- Examples
 - Blending, compaction, extrusion, biological product (fruits ..)
- Representativity of the spectral measurement

NIR Measurement Global scale



Lower scale
Spectrum of type 1 particles
Spectrum of type 2 particles

- Critical attributes
 - Physical: Particle size, density, homogeneity, structure conformity
 - Chemical: Chemical concentration, drying, adulteration





Complex Liquid Products

- Binary mixture :
 - Liquid/ solid, Liquid / Liquid, Liquid / Gaz
- Examples :
 - Fermentation, Crystallization, Dissolution
- Representativity of the spectral measurement

NIR Measurement Global scale

Critical attributes

- **Physical**: Particles or cells size and density (turbidity)
- Chemical: Chemical attributes of the suspension



Lower scale
Spectrum of particles in suspension

Spectrum of Liquid

"Real-life" measurement configuration

- The representativity of the spectra can be affected by the measurement environment
 - In lab (controlled conditions)
 - Into a reactor (liquid or solid)
 - Into tube/pipe during transfer
 - On conveyor belts
 - Through packaging







- What are "Complex products"
- Principle of Spatially Resolved Spectroscopy (SRS)
- Light/Matter interaction simulations
- Chemometrics
- INDATECH solutions
- Application examples



Spatially Resolved Spectroscopy (SRS - 1989)





Transmission Measurements



- Measurements at different distances
- Measurement in transmission and reflection
- Main benefits for :

SRS:

- Scattering products
- Heterogeneous Products,
 e.g. multi-layered/ coated products

- What are "Complex products"
- Principle of Spatially Resolved Spectroscopy (SRS)
- Light/Matter interaction simulations
- Chemometrics
- INDATECH solutions
- Application examples



Light / Matter Interaction within the product



➔ A better understanding of scattering enables the design of new tools for a better product characterization



Tools from the medical field

- Spatially Resolved Spectroscopy:
 - Many medical research centers have worked on this technique
 - Several simulation tools for light/matter interaction have been developed:
 - Monte-Carlo simulation : Millions of photons are launched.
 - ➔ A photon path is defined by product attribute and statistical/ probabilistic model



Example : change in scattering



Example : change in scattering

Monte-Carlo simulation

Absorption (μ_a =0.3 cm⁻¹), scattering (μ_s ,=5 cm⁻¹)





Example : change in scattering

Monte-Carlo simulation

Absorption (μ_a =0.3 cm⁻¹), scattering ($\mu_{s'}$ =25 cm⁻¹)

Higher scattering medias

Examples

More compaction Smaller particle size

Higher turbidity

Higher signal close to irradiation point
 BEWARE: not due to absorption change

→ More superficial / Less deep measurement



→ Tools from the medical field

- Spatially Resolved Spectroscopy:
 - Several simulation tools have been developed
 - Monte-Carlo simulation
 - Finite Elements Methods (NIRFAST)
 - The shape is modeled by pyramid
 - The radiative theory is used to calculate light absorption and scattering



UNIVERSITY^{OF} BIRMINGHAM



Pr. Hamid Dehghani NIR2013 Invited Speaker

NIR 2013 Pre-conference workshop on NIRFAST

http://www.ondalys.fr/en/nir-2013/ the 2nd of June 2013 La Grande Motte, France

 Image: Nodel :
 Shape

 Shape
 Shape

 Layers
 Chemical product

 Point of illumination
 Fesults : VIS/NIR Spectra

 Function
 Fesults : VIS/NIR Spectra

 Function
 Function

- What are "Complex products"
- Principle of Spatially Resolved Spectroscopy (SRS)
- Light/Matter interaction simulations
- Chemometrics
- INDATECH solutions
- Application examples



Layered products → Chemometrics approach



Depending on the distance, the measurement will be more sensitive to the underlying layer : coated tablet, fluid bed dryer...

The closest position provides more information from the coating



→ SRS data processing solutions:

- Subtract position 1 from the others
- Use data set of position 1 for a PCA and

subtract the subspace from the other

positions (EPO approach)



The farthest position provides more accuracy of deep layers

Heterogeneous products Chemometrics (solution patented)



- Uniformity and homogeneity is evaluated by spectral comparison.
 - Similar spectra : homogeneous

Irradiation

- Else : heterogeneous
 - ➔ Possible diagnosis of chemical or physical origins



17

Heterogeneous products → Heterogeneity index

- Comparison of SAM-Spec[®] 2 symmetrical signals (position #3 and #4).
- $x_h(\lambda) = \frac{x_3(\lambda)}{x_4(\lambda)}$ λ (nm) limits Heterogeneity index computed vs reference measured on optical standards

 $\mathbf{\Lambda}$

- Wavelength dependent \rightarrow chemical heterogeneity
- Wavelength independent but offset \rightarrow physical heterogeneity



Confidence

- What are "Complex products"
- Principle of Spatially Resolved Spectroscopy (SRS)
- Light/Matter interaction simulations
- Chemometrics
- INDATECH solutions
- Application examples



Probe design according to the application

Tablets Capsules Strips Films Seeds Multi-layered







Powders Granules Gels Foams Turbid liquids

. . .









MEAN PARTICLE SIZEHOMOGENEITYCHEMICAL
COMPOSITIONDENSITYAGGLOMERATE DETECTIONLAYER (COATING)CELL DISCRIMINATIONCELL DENSITY20

SAM-Spec[®] solution : Spatial Advanced Measurement by Spectroscopy

- Use NIR Spectrometers
 - Multiplexed spectrometer (Bruker, Thermo, ...)
 - Spectrometer with external multiplexer (ASD, Zeiss, Avantes ...)

Optical Switch DART 501



- Use hyperspectral camera: Hy-Ternity[®]
 - Detection range: 400-1000nm or 1000nm-1700nm
- Provide acquisition of 30 positions in 1-5ms





Hy-Ternity[®] : Hyperspectral Terminal



- Hyperspectral camera (400-1700nm)
- IP65 Unit, touchscreen and low voltage computer
- Compatible with all probes



Hy-Ternity[®] : Hyperspectral Terminal



- 3 independent light sources
- Temperature measurement for each probe
- Up to 3 probes simultaneously
- Detection of probe geometry



- What are "Complex products"
- Principle of Spatially Resolved Spectroscopy (SRS)
- Light/Matter interaction simulations
- Chemometrics
- INDATECH solutions
- Application examples



Detection of cracks into tablets: SAM-Spec[®] probe + Hy-Ternity[®]



Homogeneity : agglomerate detection SAM-Spec [®] probe + ASDi + DART 501 [®]

Agglomerate detection in unsieved samples





Microalgae production control Spot4Line [®] solution

- Objectives:
 - Biomass quantification : number of Microalgae cells
 - Microalgae discrimination: contamination detection
- Instrumentation:
 - Spot4line[®] : in-stream / pipeline configuration
 - 3-point SRS with a few wavelengths (UV, VIS, NIR)
- Results:











Conclusion

- Spatially Resolved Spectroscopy (SRS) :
 - Useful for complex products / highly scattering media
 - Chemical (absorption) and Physical (scattering) characteristics
 - Better sample representativity
 - Homogeneity measurement
 - In-depth/multi-layer measurements
- Practical implementation: a bridge between NIR and imaging
 - Classical VIS-NIR spectrometer with external optical switch
 - Multichannel spectrometer
 - Hyperspectral imaging sensor
 - Custom designed probes adapted to each application











Thank you for your attention!

• Contacts :

Fabien CHAUCHARD, PhD fchauchard@indatech.eu





