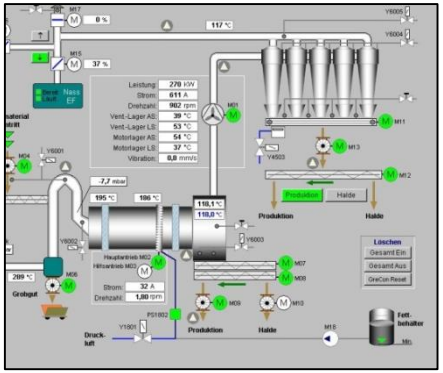

Congrès ‘Chimiométrie XX’

Study of rehydration of different organic substrates by means of near-infrared spectroscopy and signal de-mixing techniques

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BioEnTech

irstea Institut national de recherche en sciences et technologies pour l’environnement et l’agriculture

Lbe

INRA SCIENCE & IMPACT

1

Introduction & Context

1.1 Introduction and context

A large diversity of wastes for anaerobic digestion

Inputs → **Anaerobic Digestion** → **Outputs**

Bio-waste (FFOM)

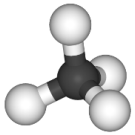
Sludge from Sewage treatment (STP)

Crop residues

Urban and industrial effluents

Animal droppings

New biomass



Biogas (CH_4) → Electricity & Heat

Digestate

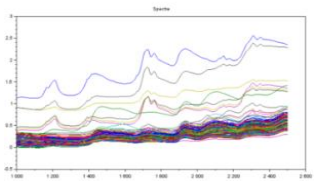
- **A need for fast substrate characterization**
- **To optimize feeding strategy**

1.1 Introduction and context

IR-SCAN® - NIRS for organic waste characterization

Substrates (organic waste)

Freeze-drying and grinding



NIR scan

Chemometrics

- Proteins
- Lipids
- Carbohydrates
- Chemical Oxygen demand (COD)
- **Methane potential (BMP)**
- CH_4 kinetics

0 → 4 days

Advantages

- **Fast measure** (4 days vs. 1 month)
- Applicable **on high diversity of wastes**
- Accuracy and **reproductibility**
- Possible to optimize feeding strategy

Hurdles

- **Sample preparation is required** to reduce effects of water and granulometry :
  - **Time-consuming step** + additional costs
  - Limits online applications

1.1 Introduction and context

Project scope

- **On-going thesis project** with BioEnTech, LBE (INRA) and ITAP (IRSTEA) to deepen **understanding of water effects** on NIRS applied to organic wastes and **find ways to correct them**

- What can we learn from **signal demixing techniques** (MCR-ALS and PARAFAC) applied to **spectra of rehydrated samples** ?

2

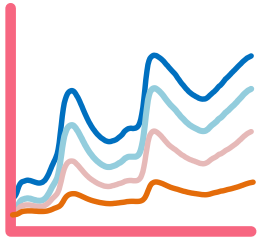
Rehydration experimental design

2.1 Rehydration experimental design

Methodology

Selection of **14 samples** (food industry wastes, digestates, crop wastes)

Building of a **range of moisture content** for each sample through rehydration (5% < DM < 99%)

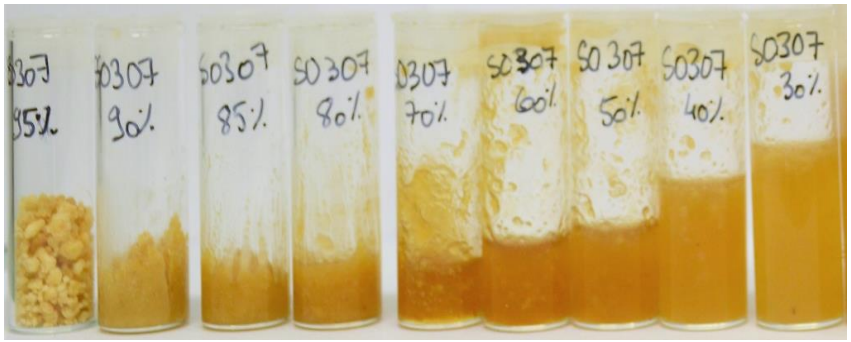


For each sample, **triplicate spectra** (with remixing) acquired using Buchi NIRFlex N-500 FT-NIR

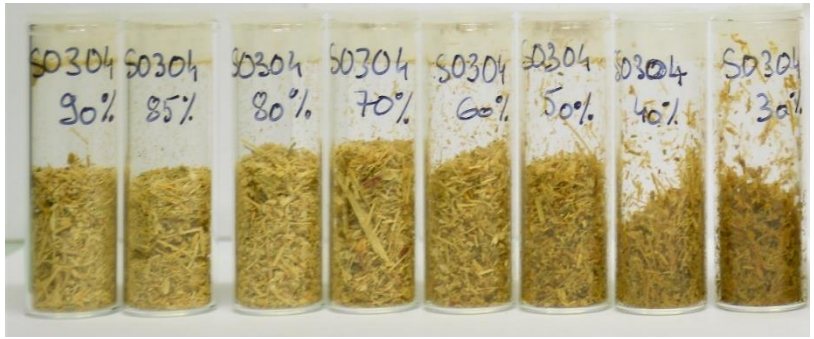
2.1 Rehydration experimental design

Methodology : some examples

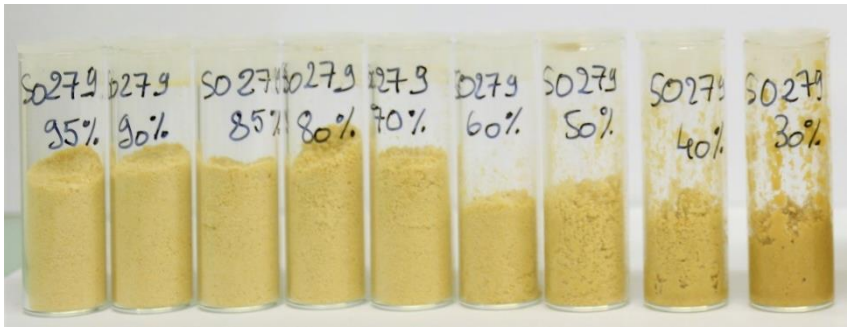
Dry Matter = 95% → **Re-hydration** → **Dry Matter = 5%**



Dried pineapple



Sorghum

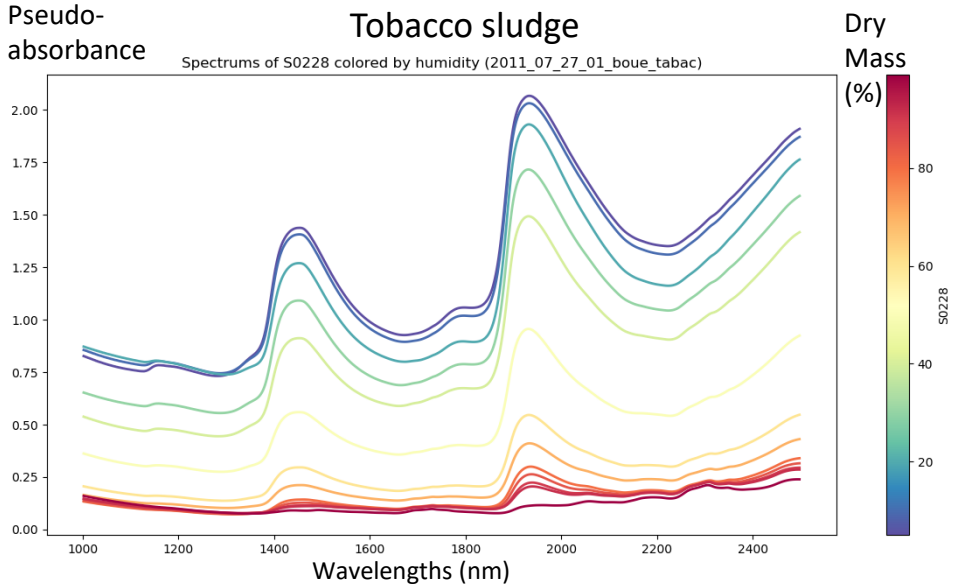


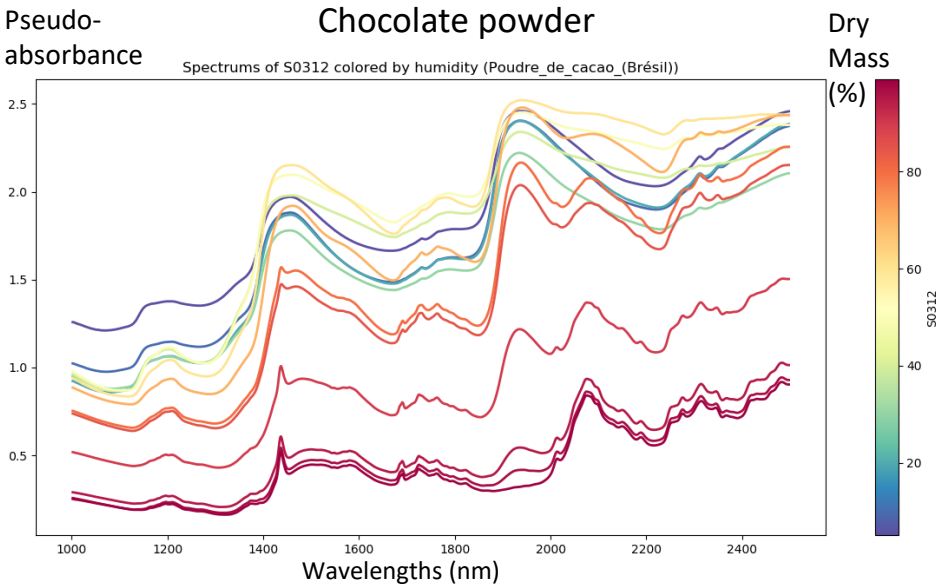
Greasy residue (IAA)

Tobacco sludge

2.1 Rehydration experimental design

Results : typology of substrates





- **Overall increase of absorption** due to changes in scattering
- **2 main water OH bands** appear
- **Substrate-specific** behaviour

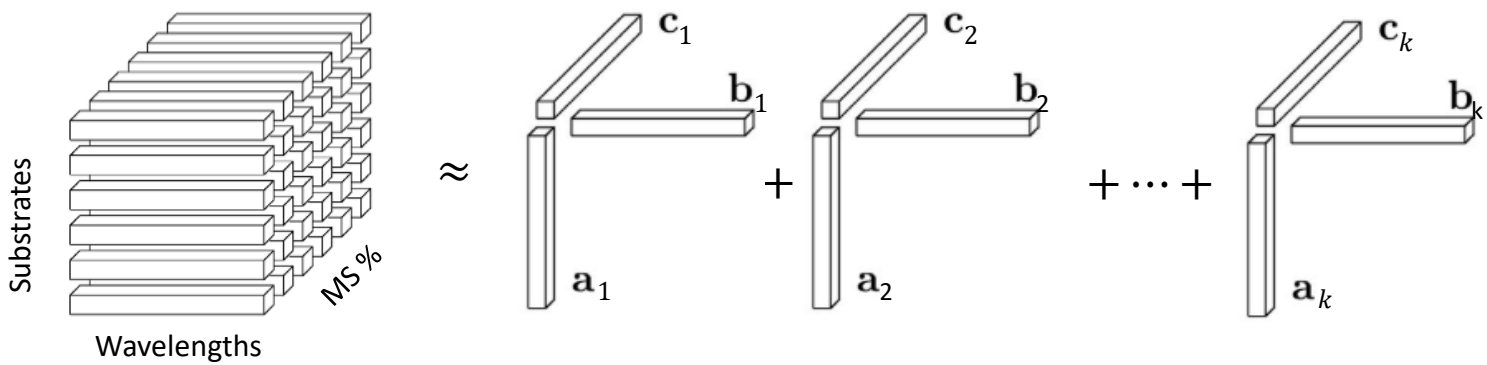
2.1 Rehydration experimental design

Methodology : signal demixing techniques

- **Principle of methods**
  - decompose a matrix or tensor of spectra X into pure spectra / components and their relative concentrations

- **1) MCR-ALS**
  - $\mathbf{X}_{(n,p)} = \mathbf{C}_{(n,k)}.\mathbf{S}_{(k,p)}^{T} + \mathbf{E}_{(n,p)}$
  - Requires initial guess of C or S (SIMPLISMA, ICA)
  - Non-nested technique implies non-direct selection of k

- **2) CANDECOMP/PARAFAC (= CP decomposition)**
  - $\boldsymbol{x}_{(n,p,q)} = \sum_{k=1}^{K} \boldsymbol{a}_{(n,k)}.\boldsymbol{b}_{(p,k)}.\boldsymbol{c}_{(q,k)} + \boldsymbol{e}_{(n,p,q)}$

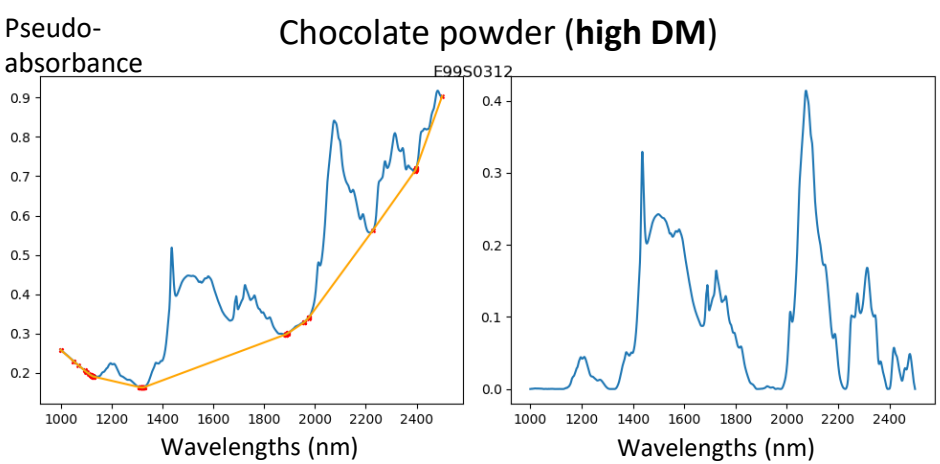


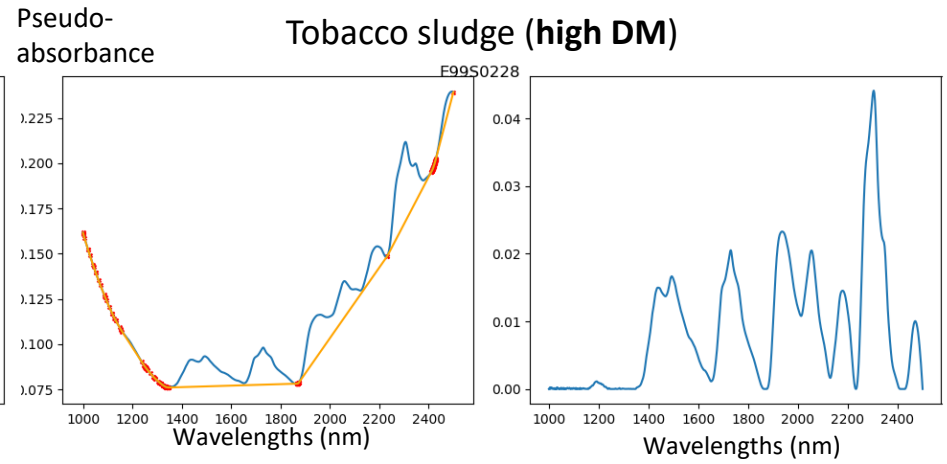
  - Non-nested technique

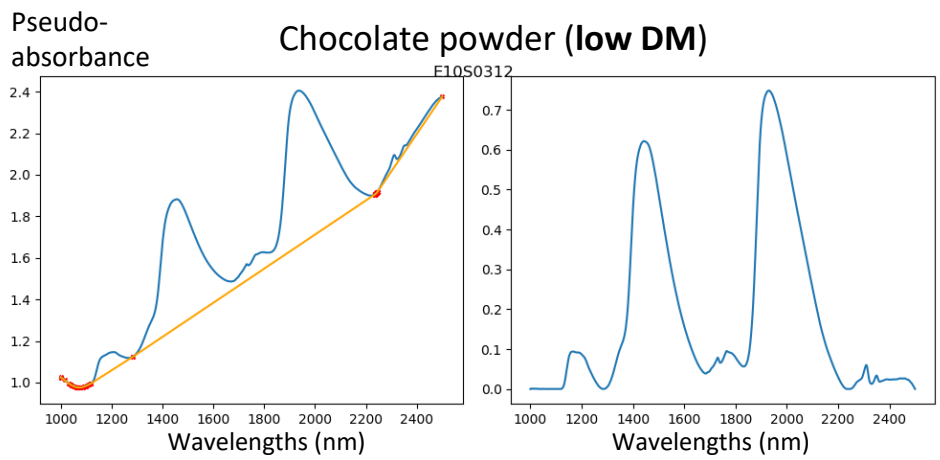
2.1 Rehydration experimental design

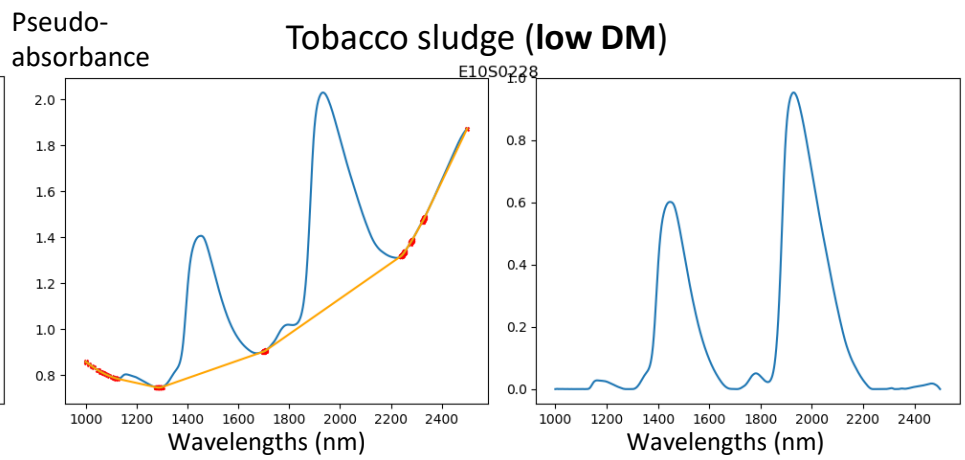
Methodology : used pretreatment

- Baseline removal with **continuum removal algorithm** (Clark and Roush (1984))





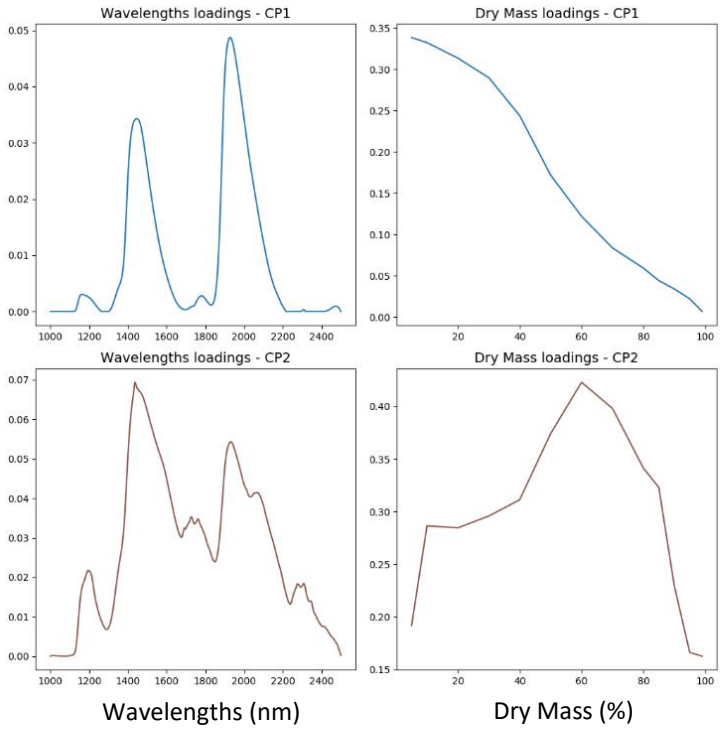


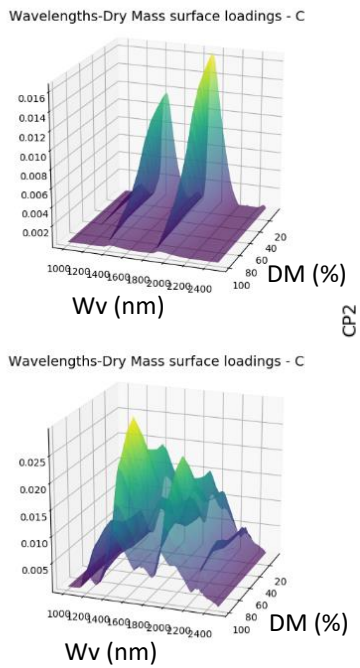


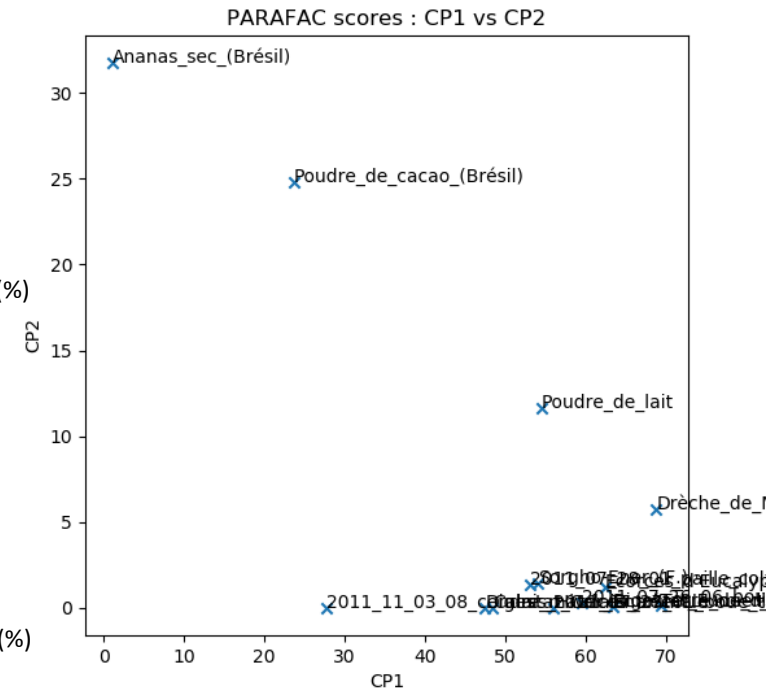
2.1 Rehydration experimental design

Results : CP decomposition

- Learnings from a **2-components model**:
  - Component 1 : **dilution of pure water spectra** with 3 main OH peaks; linear effect
  - Component 2 : **chemical interaction of carbohydrates** (carbohydrate complexity: digestate/sludge > cellulose > simple sugar) present at average range of dry mass content (30-80%)



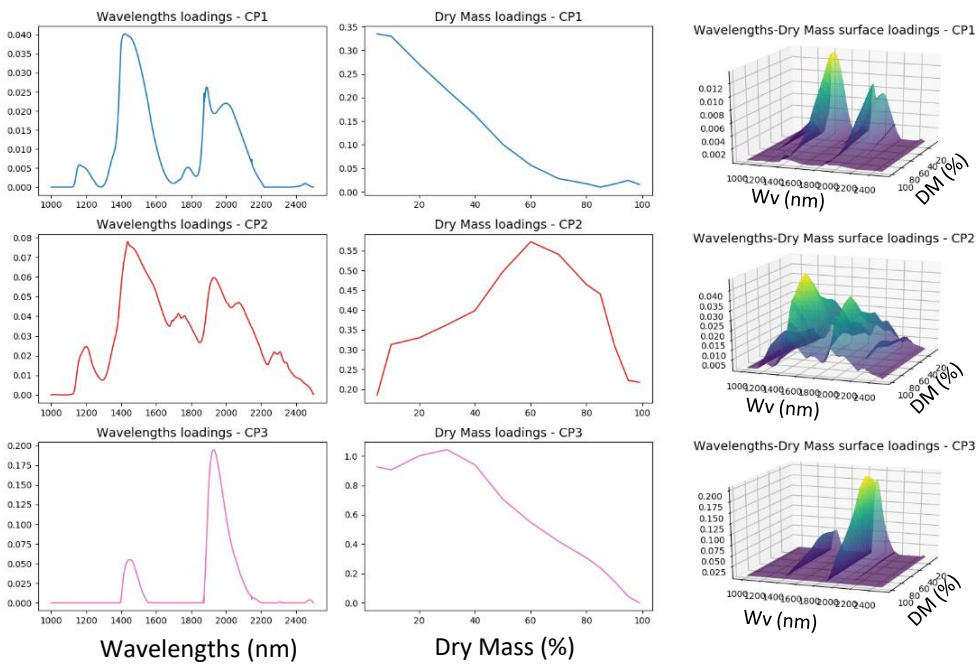


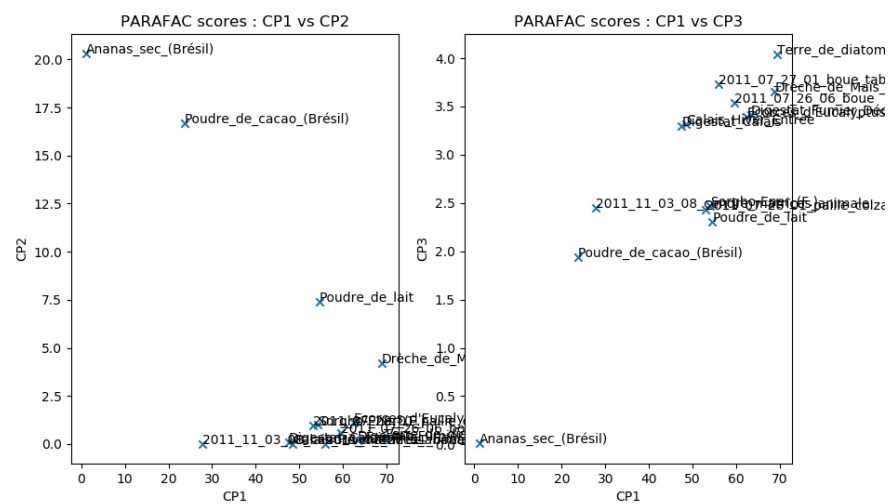


2.1 Rehydration experimental design

Results : CP decomposition

- Learnings from a 3-components model:
  - Component 2 unchanged: **chemical interaction of carbohydrates** (carbohydrate complexity: digestate/sludge > cellulose > simple sugar) present at average range of DM (30-80%)
  - Dilution is detailed in two sub-phenomenas (Component 1 and 3):
    - **pure water spectra linear dilution**
    - A **saturating OH band peak (1940-2000 nm)** below DM=40%





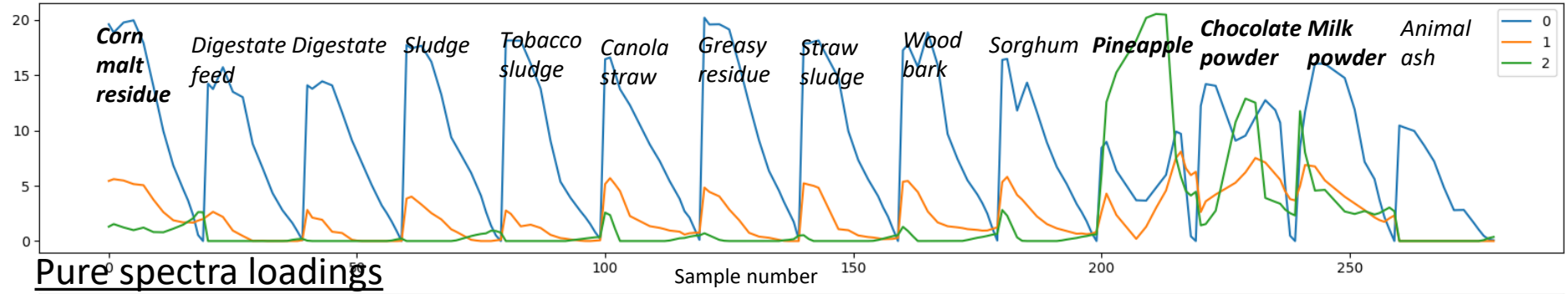
2.1 Rehydration experimental design

Results : MCR-ALS with MultiSet option

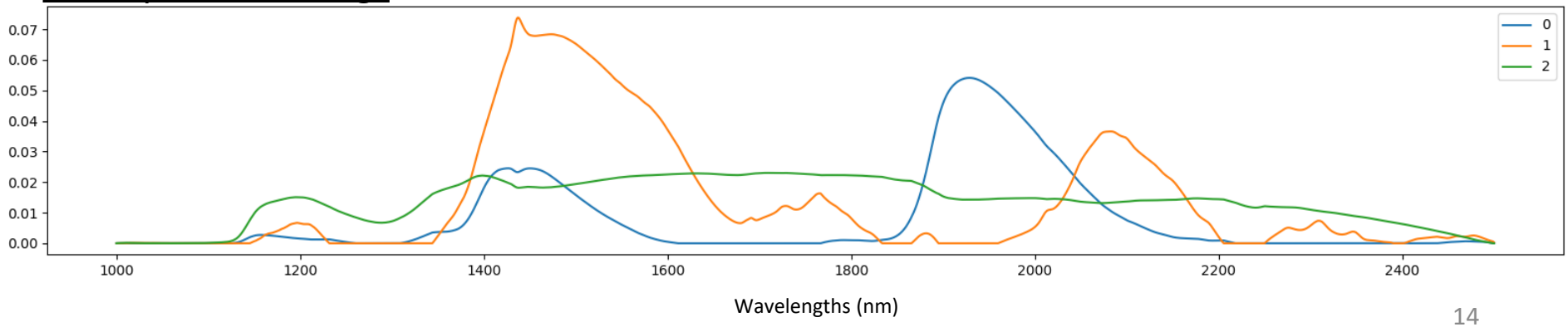
- Using MCR-ALS with multiset option :
  - **Same first component** linked to water OH bounds
  - Second and third components linked with the **interaction of water with carbohydrates and signal saturation**

Concentration profiles

Substrates rich in carbohydrates



Pure spectra loadings



3

Conclusion

3.1 Conclusion

Conclusion

Summary of results

- PARAFAC method seems most suitable for such dataset
- NIRS behaviour with water is related to complexity of carbohydrates
- Water effect evolves along moisture content :
  - At low and intermediate moisture content, we have chemical interaction
  - At high moisture content, dilution effect is dominant

Next steps / perspectives

- Consolidate database : DM resolution, number of samples
- Study dehydration instead of rehydration to investigate water states

BioEnTech

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Thank you!

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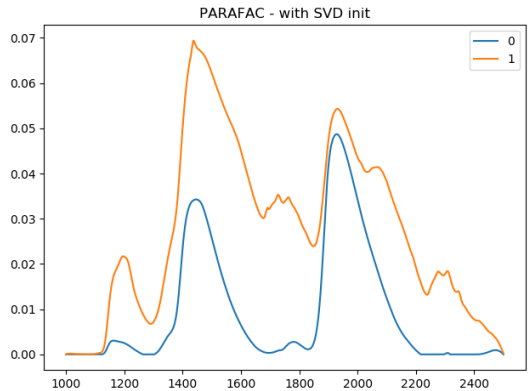
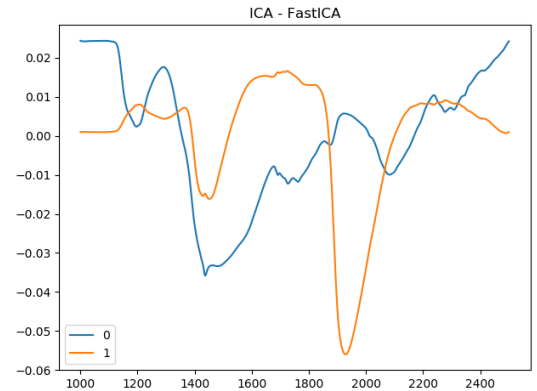
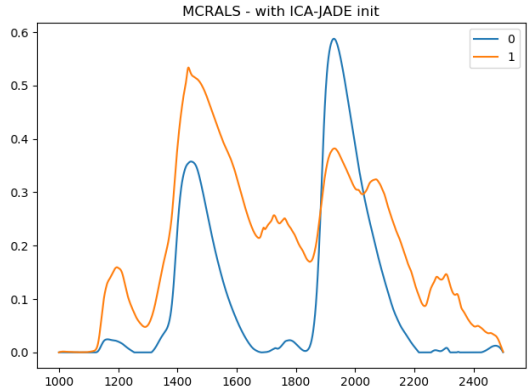
Cyrille CHARNIER

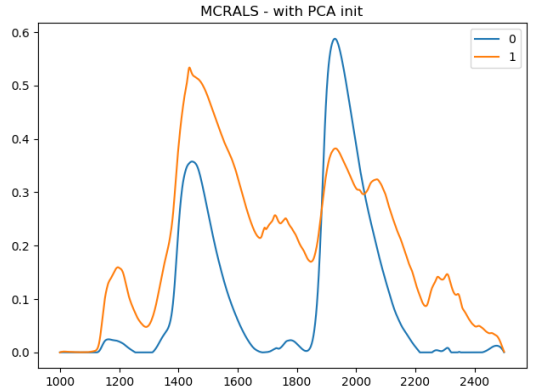
2.1 Rehydration experimental design

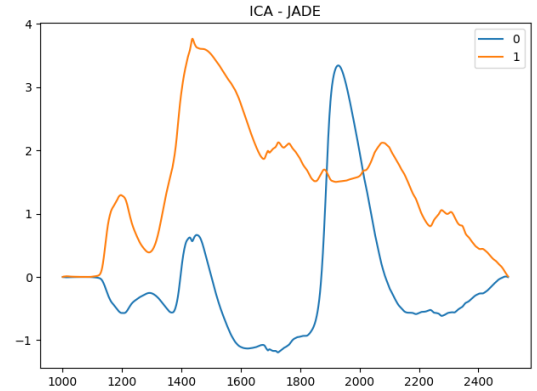
Results : Comparison of methods (2 components)

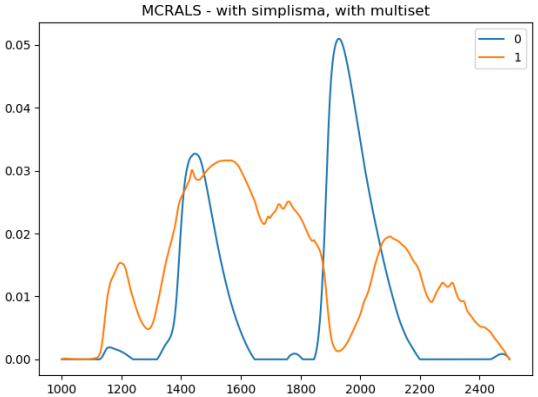








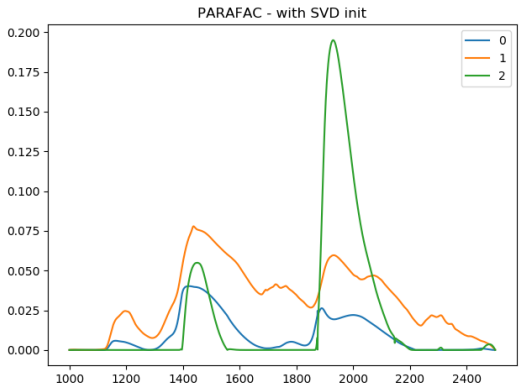


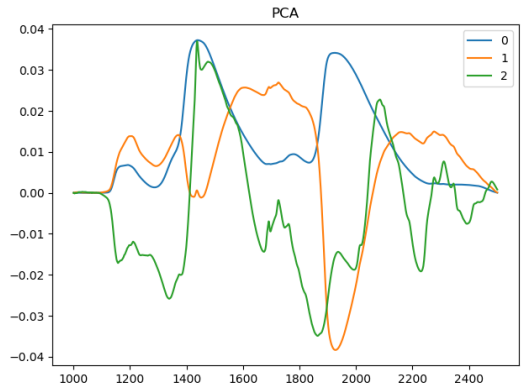


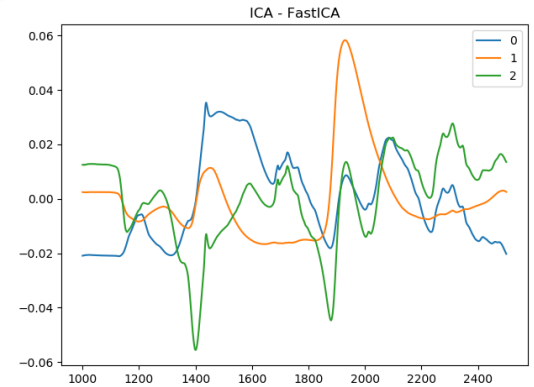
- 2-component model : all techniques provide same results except PCA (as expected!)

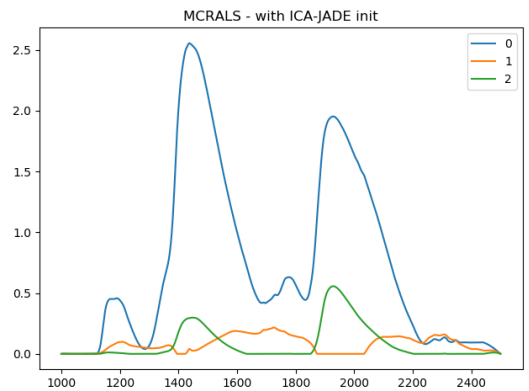
2.1 Rehydration experimental design

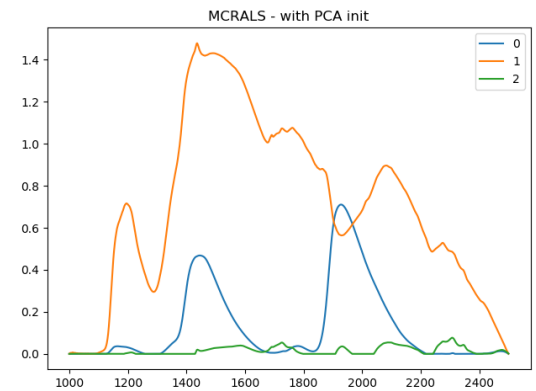
Results : Comparison of methods (3 components)

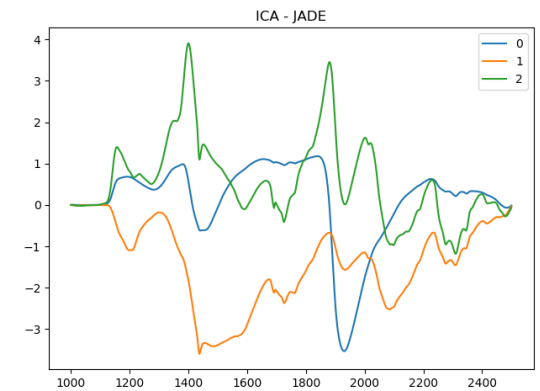


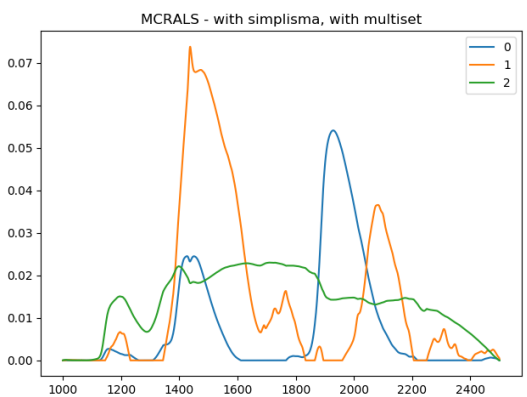












2.1 Rehydration experimental design

Results : Comparison of methods (4 components)