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

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Introduction & Context
Introduction and context

A large diversity of wastes for anaerobic digestion

- Bio-waste (FFOM)
- Sludge from Sewage treatment (STP)
- Crop residues
- Urban and industrial effluents
- Animal droppings
- New biomass

Inputs → Anaerobic Digestion → Outputs

Electricity & Heat

Biogas (CH₄)

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

**Advantages**
- Fast measure (4 days vs. 1 month)
- Applicable on high diversity of wastes
- Accuracy and reproducibility
- 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 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%**: Dried pineapple
- **Re-hydration**
- **Dry Matter = 5%**: Sorghum

- **Dry Matter = 95%**: Greasy residue (IAA)
- **Re-hydration**
- **Dry Matter = 5%**: 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
Principle of methods
- decompose a matrix or tensor of spectra $X$ into pure spectra / components and their relative concentrations

1) MCR-ALS
- $X_{(n,p)} = C_{(n,k)} \cdot S_{(k,p)}^T + E_{(n,p)}$
- Requires initial guess of $C$ or $S$ (SIMPLISMA, ICA)
- Non-nested technique implies non-direct selection of $k$

2) CANDECOMP/PARAFAC (= CP decomposition)
- $x_{(n,p,q)} = \sum_{k=1}^{K} a_{(n,k)} \cdot b_{(p,k)} \cdot c_{(q,k)} + e_{(n,p,q)}$
- Non-nested technique

Substrates
\[\text{Wavelengths} \approx c_1 + c_2 + \cdots + c_k \]

Non-nested technique
Rehydration experimental design

Methodology: used pretreatment

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

### Wavelengths (nm) Pseudo-absorbance

**Chocolate powder (high DM)**

**Tobacco sludge (high DM)**

**Chocolate powder (low DM)**

**Tobacco sludge (low DM)**
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%)
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%
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**

**Pure spectra loadings**

**Substrates rich in carbohydrates**
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
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Thank you!

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Rehydration experimental design

Results: Comparison of methods (2 components)

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

Results: Comparison of methods (3 components)
2.1 Rehydration experimental design

Results: Comparison of methods (4 components)