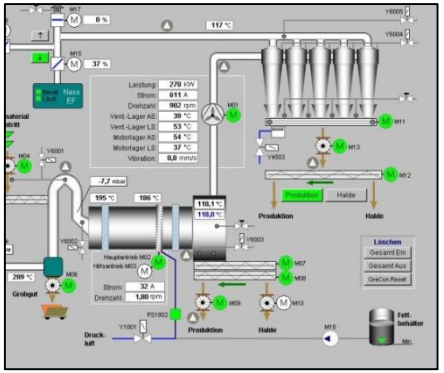
# Rencontres HELIOSPIR 2018



## Robust near-infrared spectroscopic characterization of wet organic wastes : how to avoid water interferences ?

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BioEnTech

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Lbe

INRA SCIENCE & IMPACT

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# Introduction & Context

# 1.1 Introduction and context

## A large diversity of wastes for anaerobic digestion

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

Bio-waste (FFOM)

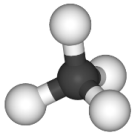
Sludge from Sewage treatment (STP)

Crop residues

Urban and industrial effluents

Animal droppings

New biomass



Biogas ($CH_4$)

Electricity & Heat

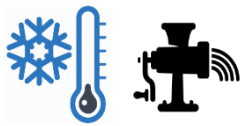
Digestate

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

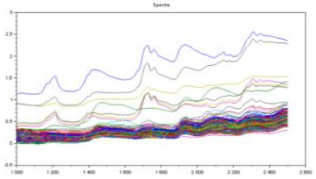
# 1.1 Introduction and context

## 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

## History of NIRS for organic waste characterization

- **Previous works at LBE/ITAP :**

NIRS prediction of BMP on Municipal Solid wastes (Lesteur et al. 2011) → Co-development of FlashBMP® (Ondalys, Veolia, LBE) → NIRS prediction of BMP, carbohydrates, proteins, lipids, COD, kinetics (Charnier et al. 2015)

- New **on-going thesis project** with BioEnTech, LBE (INRA) and ITAP (IRSTEA) to deepen **understanding of water effect** on NIRS applied to organic wastes and **find ways to avoid it**

- **First experimental results from an internship** at LBE with two objectives :
  ***1) Evaluate effect of water on current FlashBMP® model***
  ***2) Investigate a correction strategy***

# 1.1 Introduction and context

## What we know

- **3 main peaks attributed to water** (maxima at 1190, 1450, 1940 nm), with attributions to water's vibrational modes not agreed on

- **Impact of water was highlighted in many contexts** :
  - Soil organic carbon determination *(Sudduth & Hummel, 1993, Reeves et al. 2010, Knadel et al. 2014)*
  - Food analysis *(Büning-Pfaue 2003)*
  - COD in activated sludge *(Sarraguca et al., 2009)*
  - BMP of energetic crops *(Godin, Mayer, 2015)*

- Recent studies in soil spectroscopy suggest that **EPO successfully removed soil moisture effect** *(Minasny et al. 2011, Ge et al. 2014, Ji et al. 2015, Ackerson et al. 2015, Wijewardane et al. 2016)*

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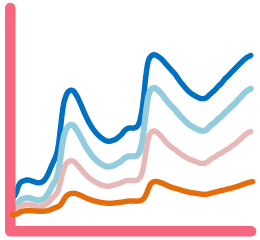
# Rehydration experimental design

# 2.1 Rehydration experimental design

## Methodology

Selection of **13 representative samples** of the FlashBMP® database (food industry wastes, digestates, crop wastes) **with known BMP**

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

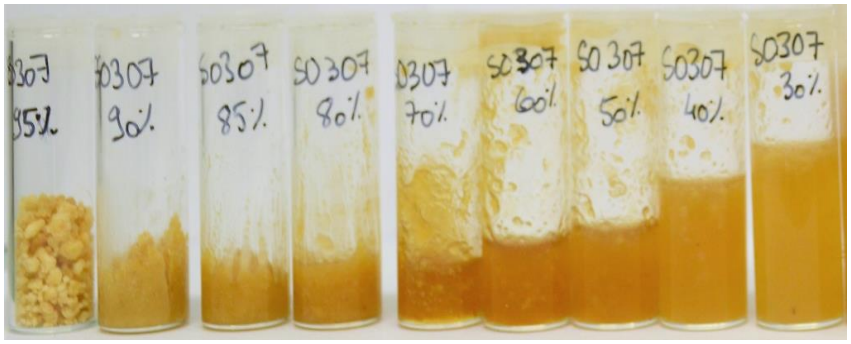


For each sample, **triplicate spectra** (with remixing) acquired using Buchi NIRFlex N-500 FT-NIR

# 2.1 Rehydration experimental design

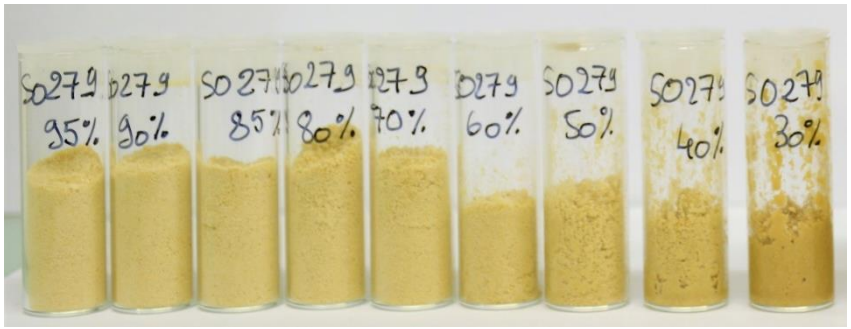
## Methodology : some examples

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

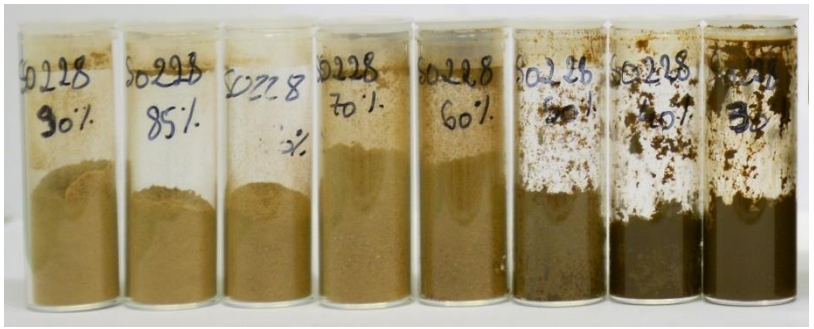


Dried pineapple

Sorghum



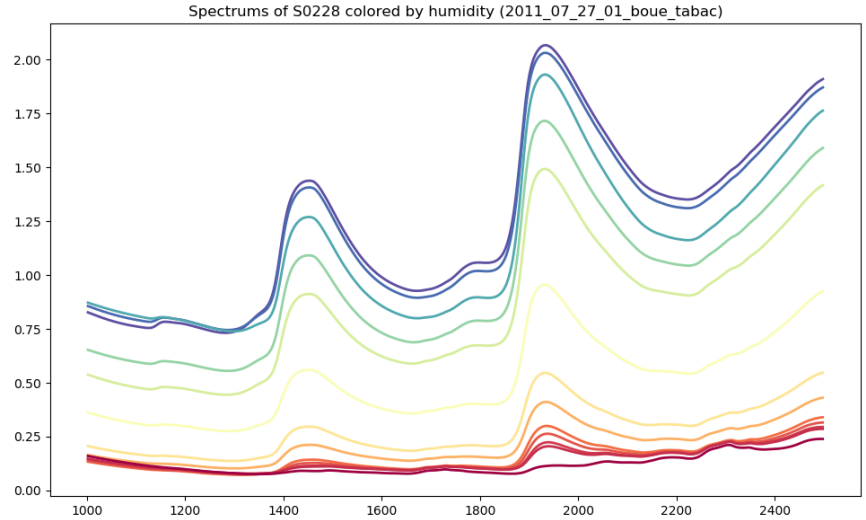
Greasy residue (IAA)

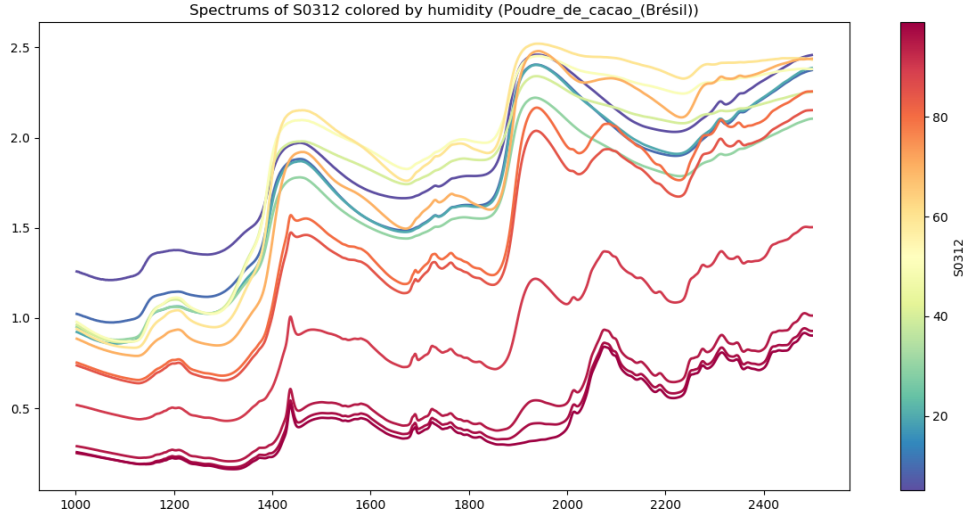


Tobacco sludge

# 2.1 Rehydration experimental design

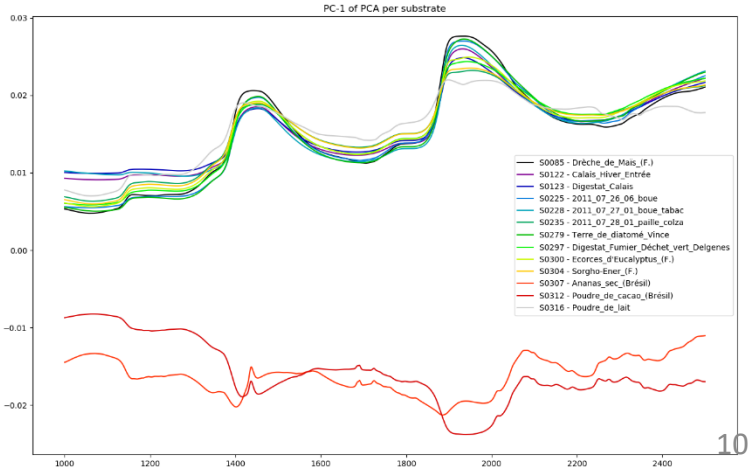
## Results : typology of substrates



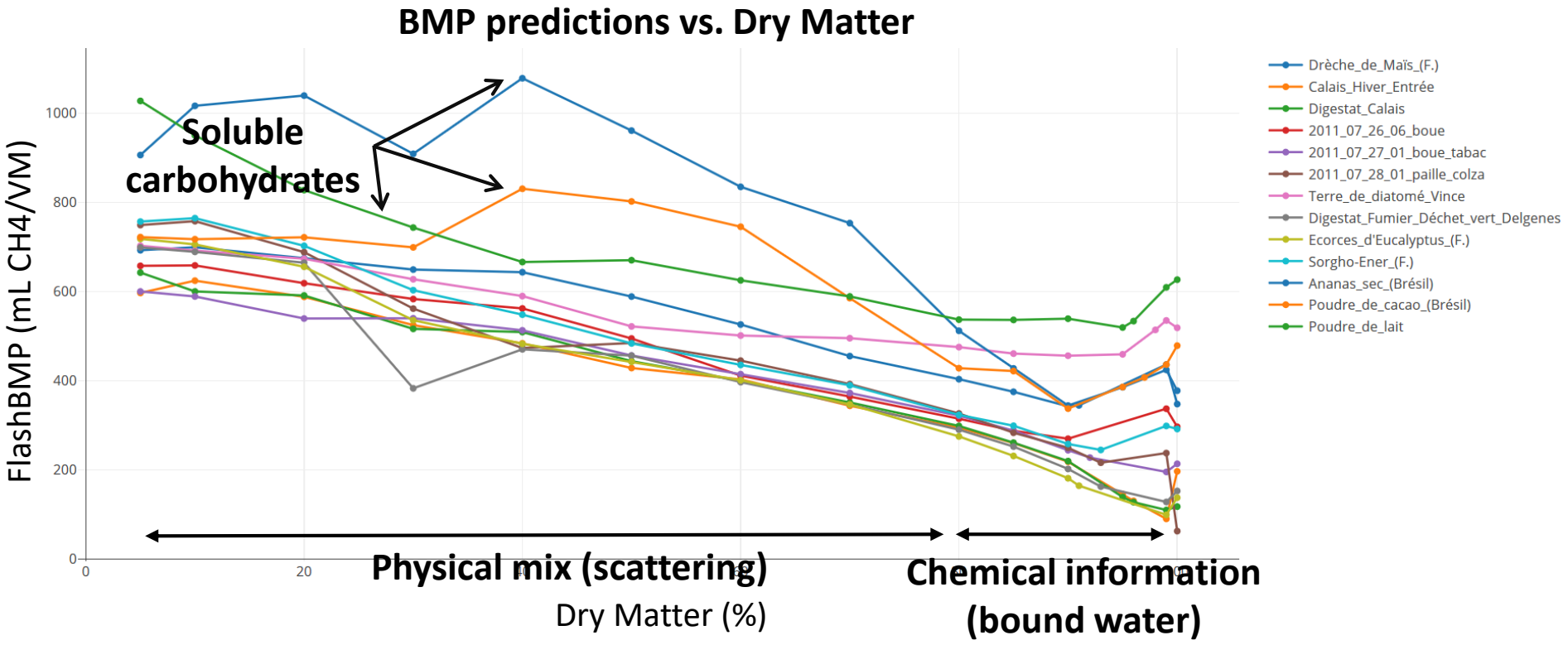

- **Carbohydrates' footprint still visible at high moisture content** (70%) compared to others

- One can observe this by looking at first **loadings of PCA per sample**



# 2.1 Rehydration experimental design

## Results : strong effects on FlashBMP® predictions




- Current model is **highly affected by water** for DM% below 90%
- **Substrate-specific effect** : water-soluble substrate types vs other

# 2.2 Rehydration experimental design

## Methodology : Model correction (update/EPO)

**FlashBMP database**

251

Random train test split (30%)

175

76

**Humid database**

13

x14 DM per sample

Random train test split (40%)

7

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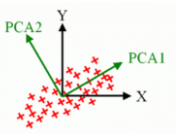
Test set

Train test

Pretreatments (SG1/SNV)

**EPO**

Nuisance space definition (PCA)




Orthogonal projection of train test to nuisance space

175

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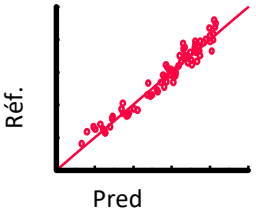
**Model update**

Repeated 5-fold CV

Determination of #LV based on min RMSECV

Model training (train test)

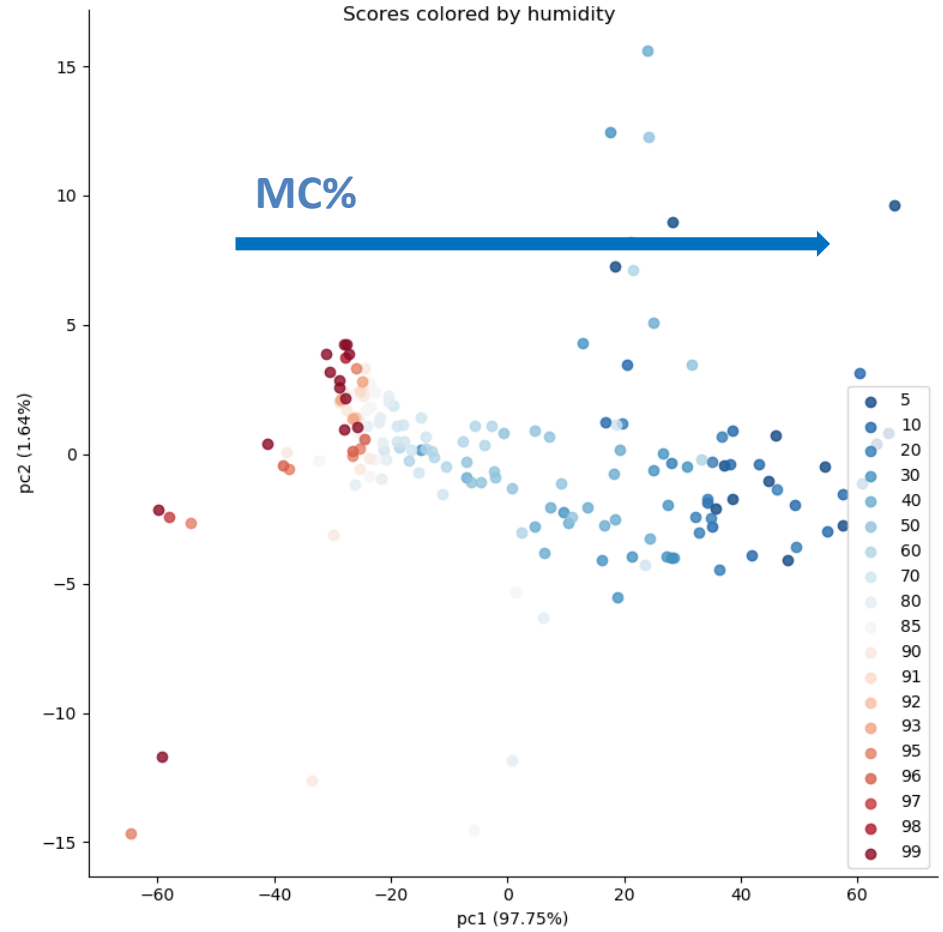
RMSEP calculation on test set (global, dry, wet)

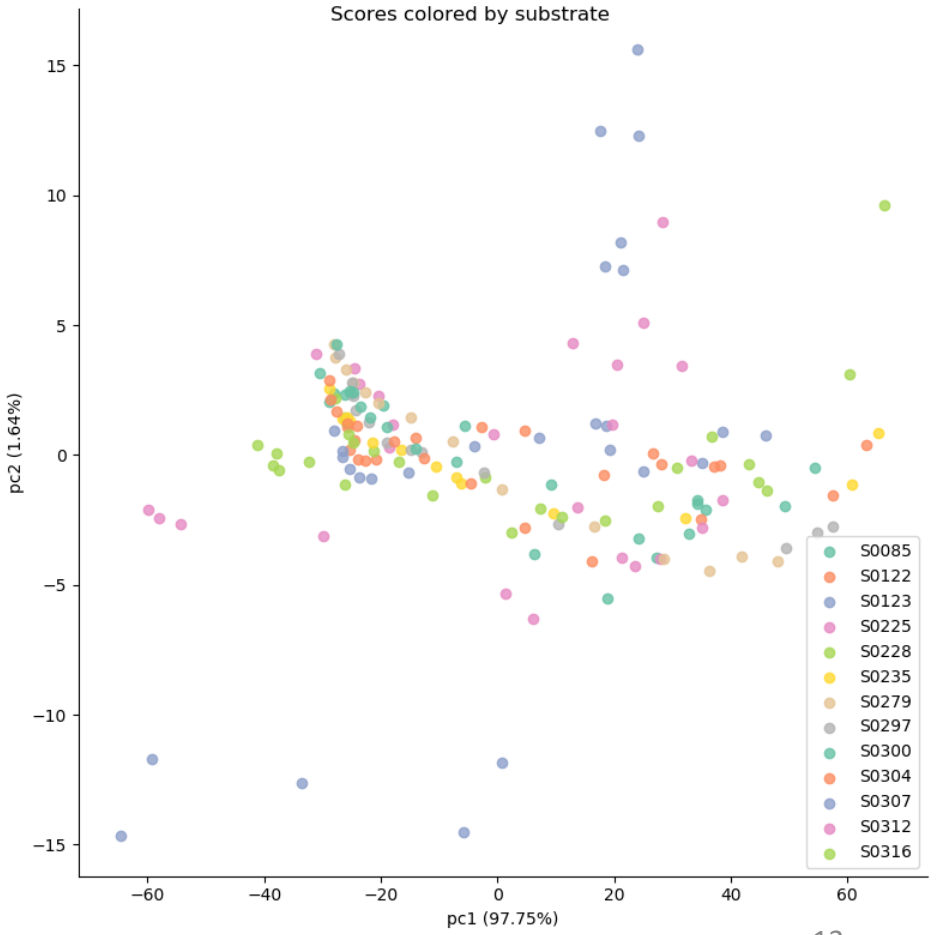


# 2.2 Rehydration experimental design

## Results : Model correction (update/EPO)

EPO : 1st and 2nd principal components (PC) catch well moisture content variations

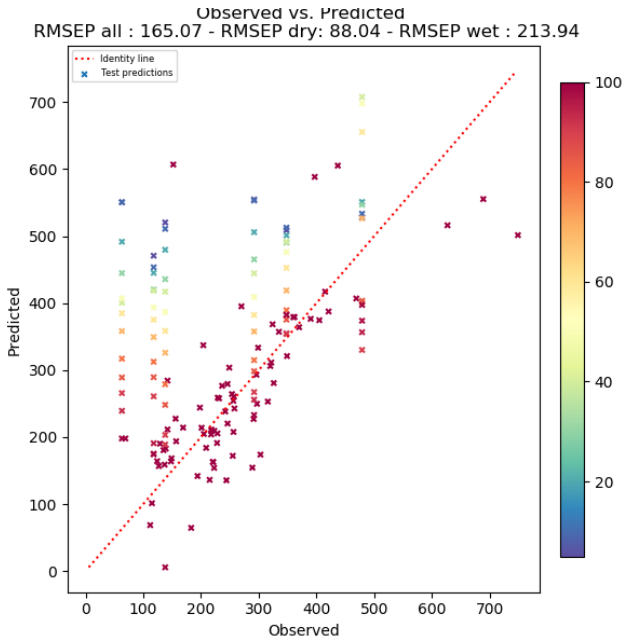


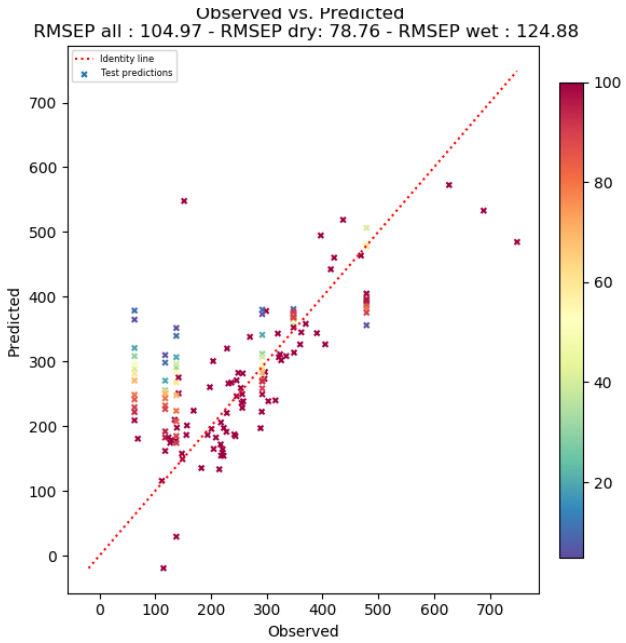
# 2.2 Rehydration experimental design

## Results : Model correction (update/EPO)

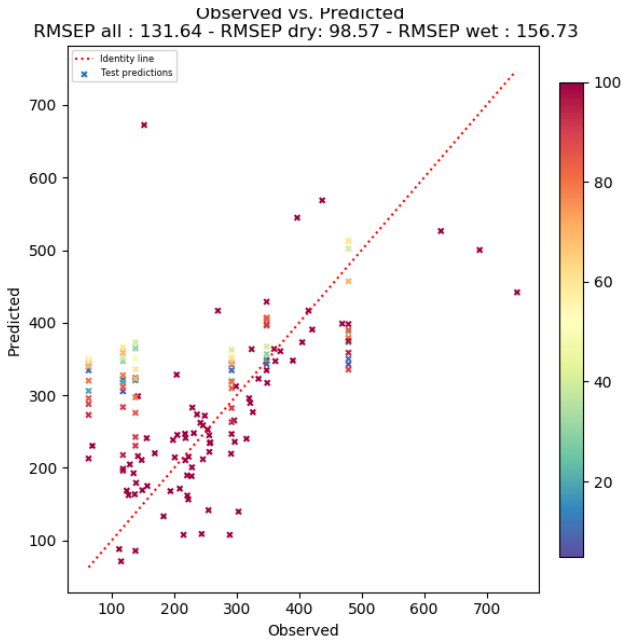
Original Model




Update Model




EPO Model




- **Model update and EPO both successful** compared to original model
- **EPO less accurate than model update**
  - wide range of wastes vs. limited nuisance database
  - wide range of moisture content
  - instability of fBMP model (complexity of BMP)

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# Conclusion

# 3.1 Conclusion

Conclusion

**Summary of results**

- FlashBMP predictions are strongly affected by water content
- Typology of substrates with carbohydrates (highly soluble substrates) => water states to be investigated
- Promising correction by update
- Correction by EPO in the current conditions was not concluant

**Perspectives**

- Test update and EPO with increased nuisance data, and reduced moisture content range
- Study dehydration vs. hydration
- Mid-term : Develop at-line and on-line applications

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# Thank you!

Alexandre MALLET, Bastien ZENNARO, Eric LATRILLE, Jean-Philippe STEYER, Ryad BENDOULA, Jean-Michel ROGER, Cyrille CHARNIER