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Analyse multi-bloc pour le suivi de la méthanisation

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INTRODUCTION

Organic waste and effluents

Biogas

Digestate

METHANIZATION PROCESS

Introduction

Temperature

O_2

pH

Ammonia

Volatile fatty acids (VFA)

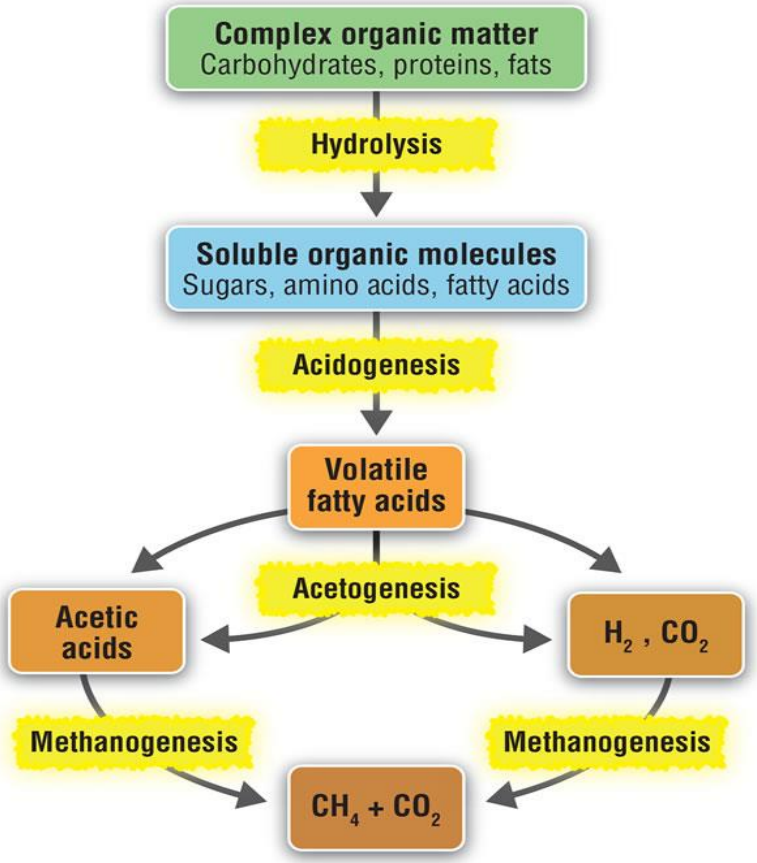
Long chain fatty acids (LCFA)

H_2

Others

Direct effect

Figure 1. Biological process of anaerobic digestion



INTRODUCTION

T°C ← Temperature sensor

O_2 ← O2 or ORP sensor

pH ← pH sensor

NH_3

LCFA ?

VFA

H_2

Others ?

TIC/Alk

CH_4/CO_2

Titrimetry *(+ conductimetry)*

Gas analyzer

+

Biogas flowmeter

- Non-monitored parameters
- Efficiency of the used technology

Introduction

- A potential solution:

Infrared Spectroscopy Sensors

Monitoring tools

Continuous and in-line	several indicators	Fast and Predictive

State of the art

Near infrared spectroscopy (NIRS)

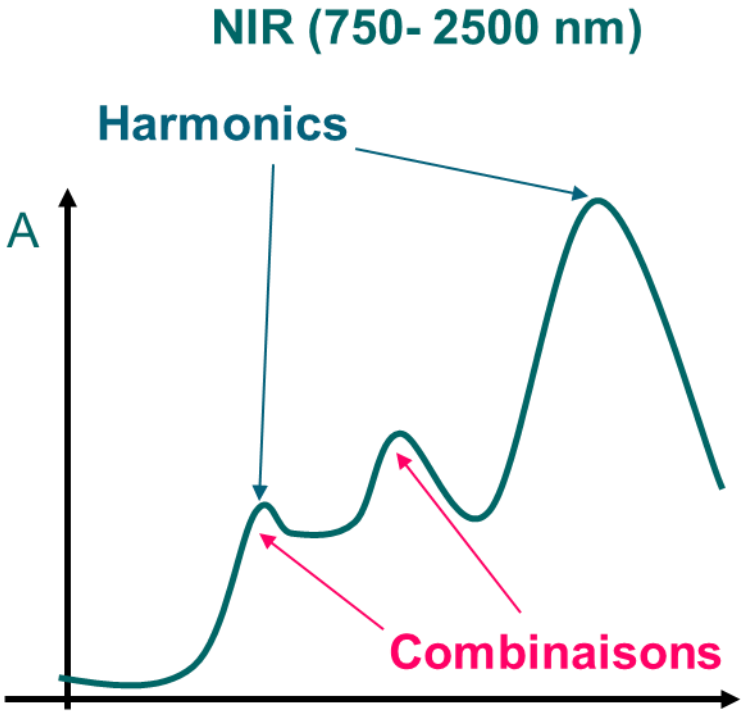
Some NIRS performances in anaerobic digestion

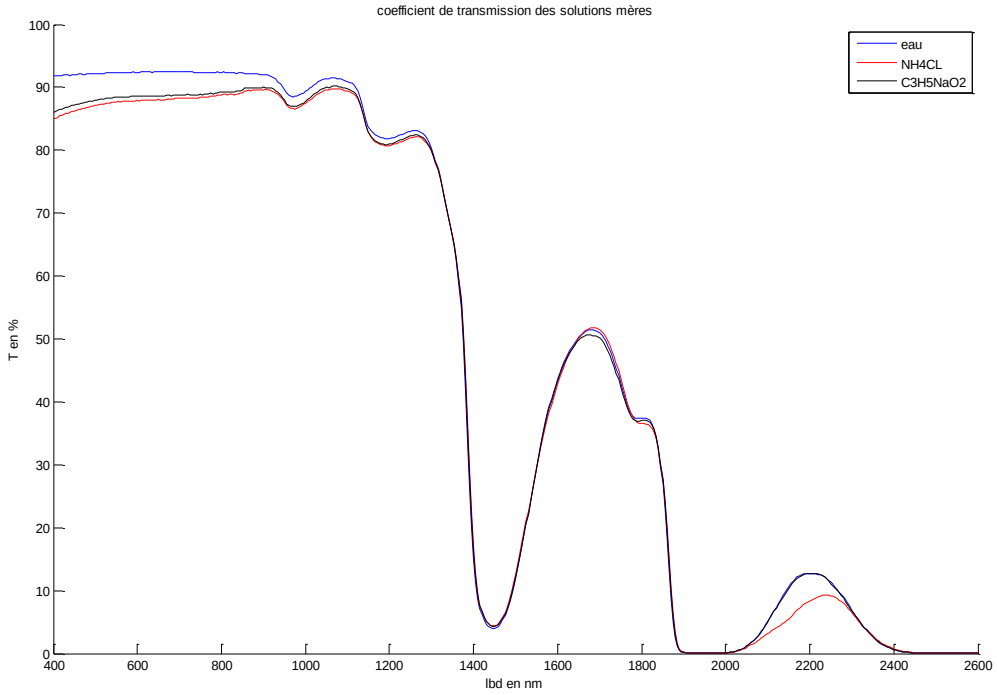
Advantages of a multi-block approach

- Near infrared Spectroscopy (NIRS)
- Some NIRS performances in anaerobic digestion
- Advantages of a multi-block approach

STATE OF THE ART

Spectroscopy: light / matter interaction





➢ Interpretation difficulties

➢ Water influence in the NIR range

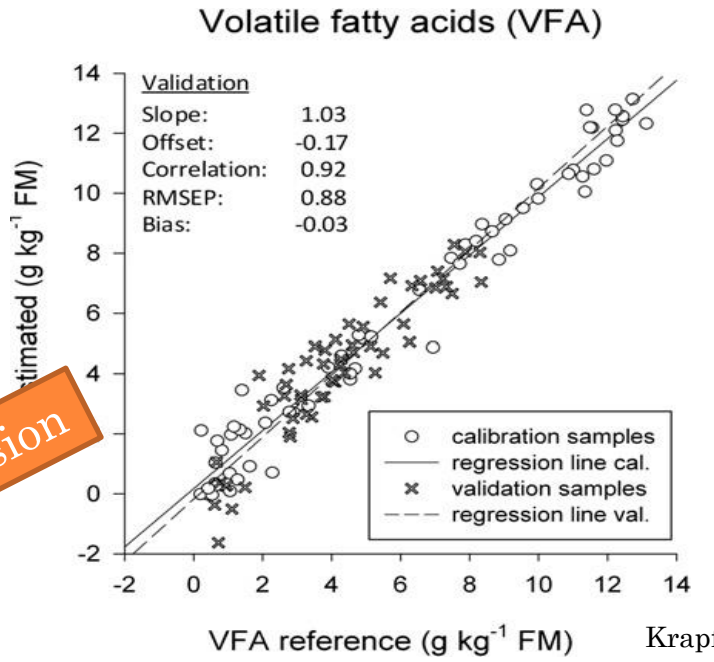
STATE OF THE ART

- Near infrared Spectroscopy (NIRS)
- Some NIRS performances in anaerobic digestion
- Advantages of a multi-block approach

Mostly on dry samples:

- pH and redox
- Total and volatile Solids
- VFA, ethanol
- Alkalinity
- Chemical oxygen demand
- Ammonia /ammonium
- Biomethane potential
- …

PLS regression

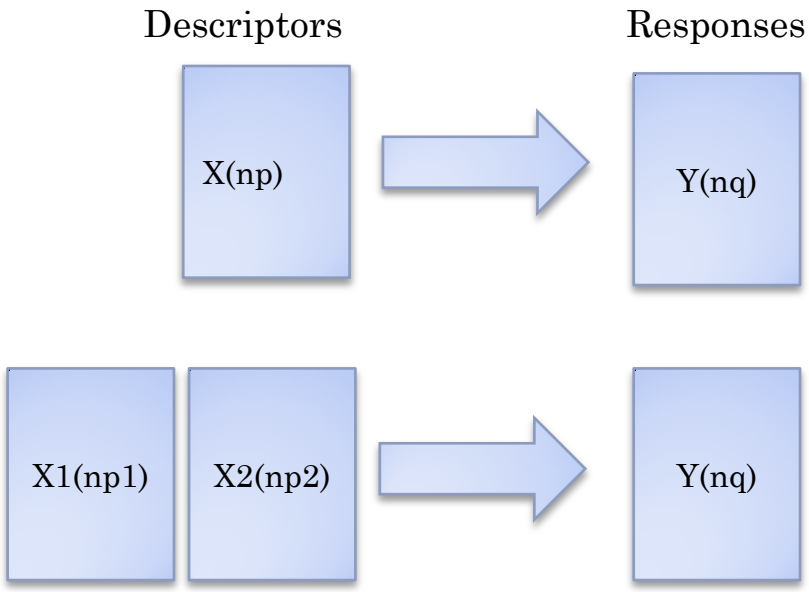


Krapf et al. 2013

References	Wavelengths (nm)	Raw digestates	R2	RMSEP (mg/kg)
Holm-Nielsen et al., 2007	960–1600	bio slurry (frozen samples)	0,84	200
Jacobi et al., 2009	850–1650	Slurry and maize silage	0,94	820
Lomborg et al., 2009	947 –1533	manure and maize silage	0,9	1590
Krapf et al., 2013	400–1750	Maize silage	0,92	900

- Near infrared Spectroscopy (NIRS)
- Some NIRS performances in anaerobic digestion
- Advantages of a multi-block approach

State of the art



- Hierarchical-PLS ,
- Multi-Block-PLS ,
- Predictive-ComDim
- Sequential and Orthogonalized Partial Least Squares (SO-PLS),
- …

- Contribution from each of the descriptors
- Selection of relevant descriptors
- Possible improvement of the prediction
- Complexity of the interpretation of latent variables

Objective

Implement a quality measurement method in NIRS on raw digestates with a multi-block analysis on spectral data and field parameters.

Methodology

Experimental setup

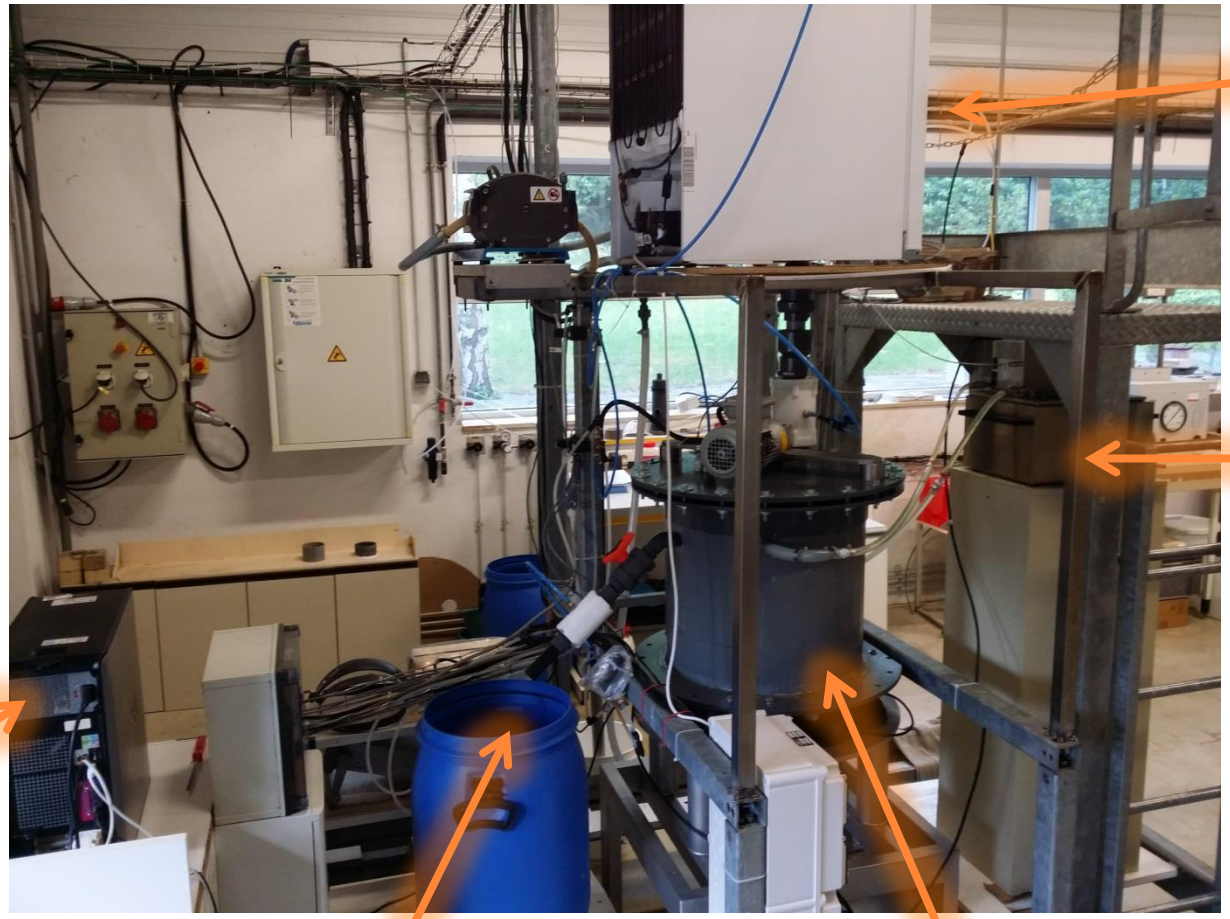
Monitored parameters

NIRS tested probes

Multi block data set

Methodology

- Experimental setup
- Monitored parameters
- NIRS tested probes
- Multi block data set



Hopper in the refrigerator

Thermostat

Acquisition under Labview

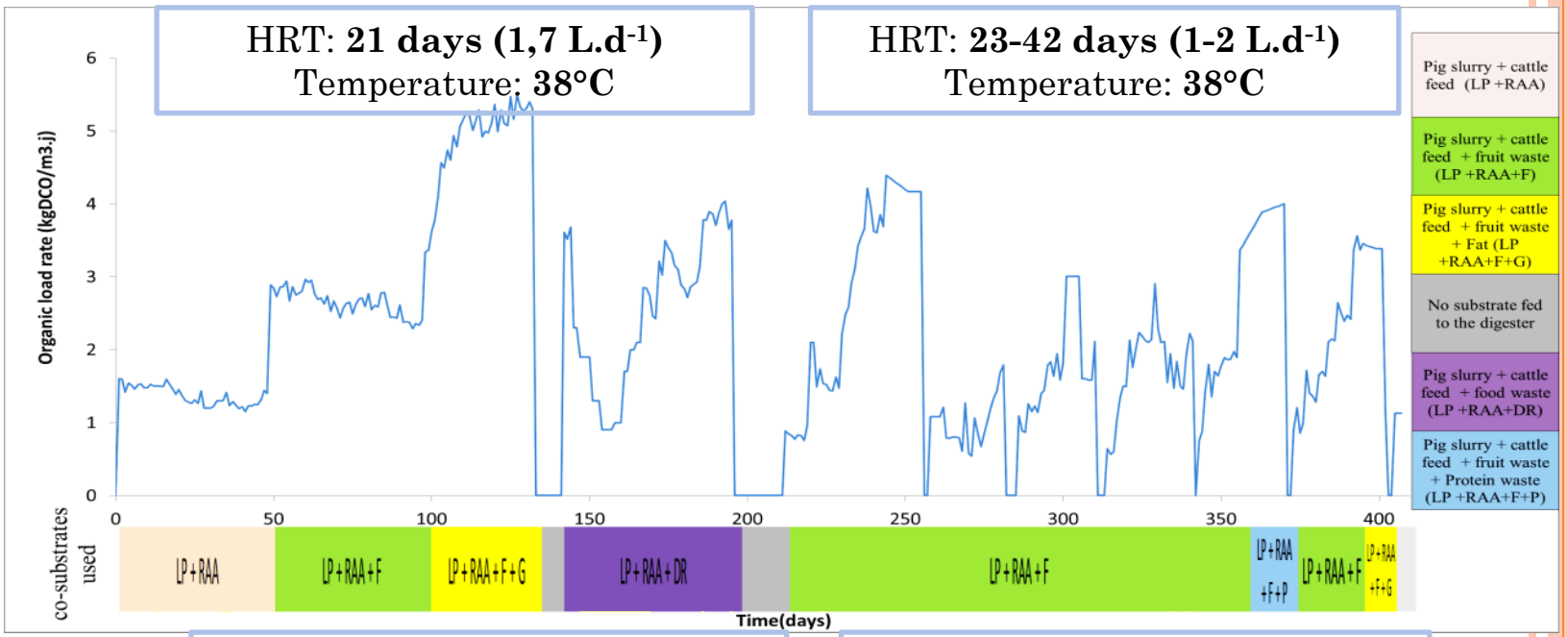
Sampling point

Continous Stirred Tank Reactor

- Experimental setup
- Monitored parameters
- NIRS tested probes
- Multi block data set

METHODOLOGY

Parameters
pH
Alk_t
TS / VS / MM
DCO
NTK, NH4+
VFAs
LCFAs
Q_{biogaz}
PCH_4 / CO_2 / H_2
…



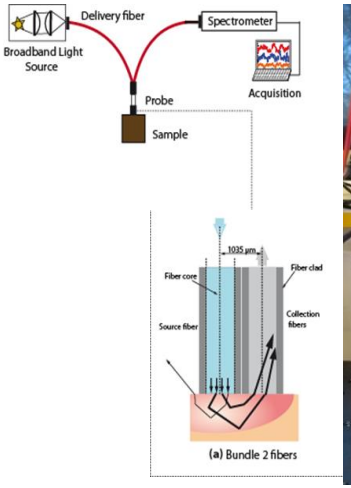
1-2 measures/week

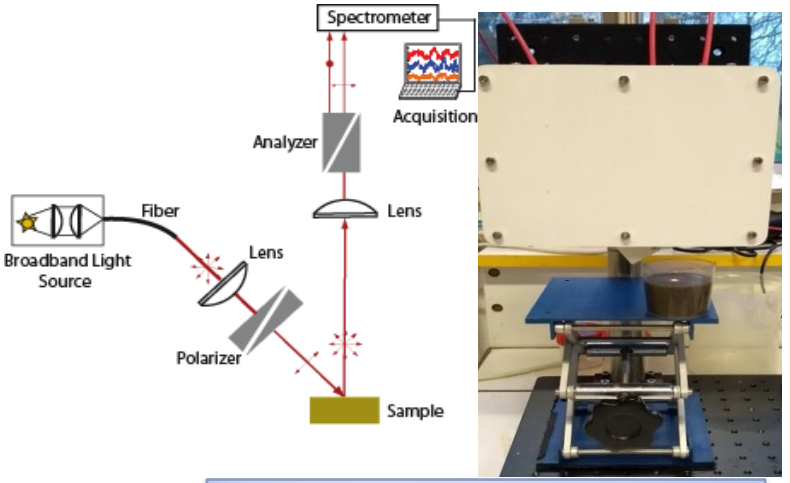
5-7 measures /week

- Pig slurry (LP)
- Cattle feed (RAA)
- Fruit waste (F)
- Fat (G)
- Food waste (DR)
- Protein waste (P)

- Experimental setup
- Monitored parameters
- NIRS tested probes
- Multi block data set

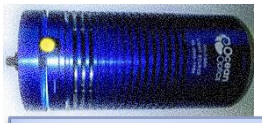
METHODOLOGY





- Two fibers: a transmitter and a receiver;
- Measurement in contact with the sample

- Measurement without contact with the sample,
- Decomposition of the total signal into polarized (single scattering) and depolarized (multi-scattering) signals.



Spectrometer and light source

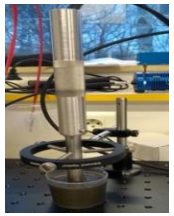
- ✓ **350-2500 nm**
- ✓ **off line measure**
- ✓ **2-7 measures /week**

METHODOLOGY

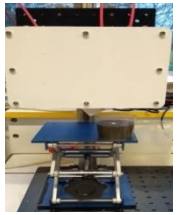
- Experimental setup
- Monitored parameters
- NIRS tested probes
- Multi block data set

Descriptors block:

Spectra X1(np)

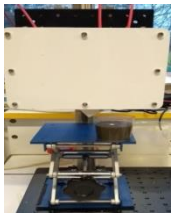


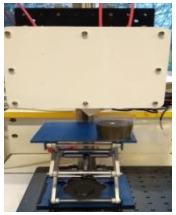
Spectra X2(np)



9 parameters X3(np)

Spectra X1(np) , X2 (np)





X3 (np) X2 (np) X1 (np)

PLS regression

Feed mass, pH in/out, Alk out, NH4+ in/out, TS&VS in and MM in.

SO-PLS : $Y = X_1B + X_2C + E$

- ✓ Fitting of Y to X_1,
- ✓ Orthogonalisation of X_2 with respect to the extracted PLS components
- ✓ Fitting the estimated residuals to X_2
- ✓ Compute the full predictive model

Næs et al., 2011

Responses block

Venitian blind (10 blocs) cross validation

Y1(nq)	**Y2(nq)**	**Y3(nq)**
Total VFA	Total LCFA	PCH4

Results and discussion

Global stability of the digester

NIRS Spectra and pretreatments

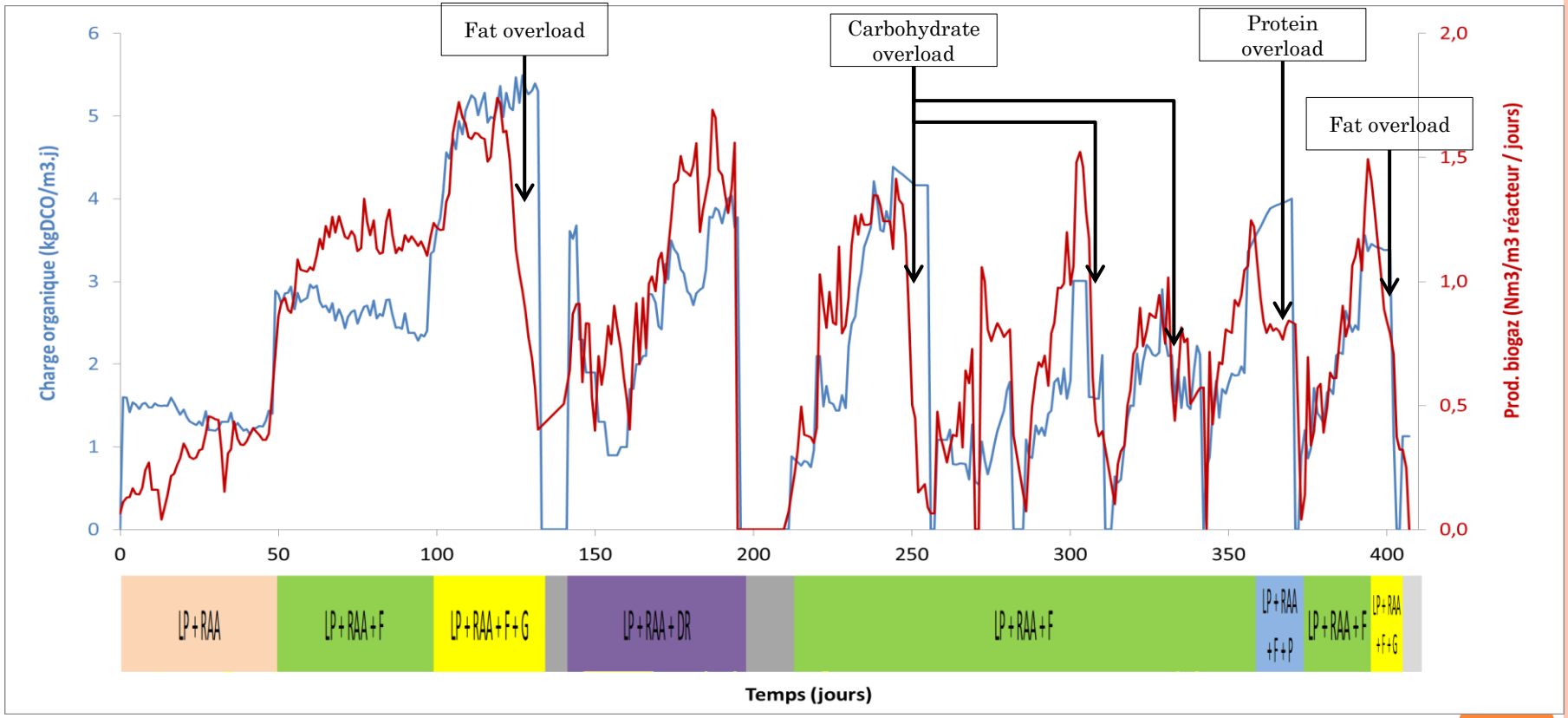
PLS regression vs SO-PLS regression

- Global stability of the digester
- NIRS Spectra and pretreatments
- PLS regression vs SO-PLS regression

RESULTS DISCUSSION

HRT: **21 days (1,7 L.d^{-1})**
Temperature: **38°C**

HRT: **23-42 days (1-2 L.d^{-1})**
Temperature: **38°C**



- Global stability of the digester
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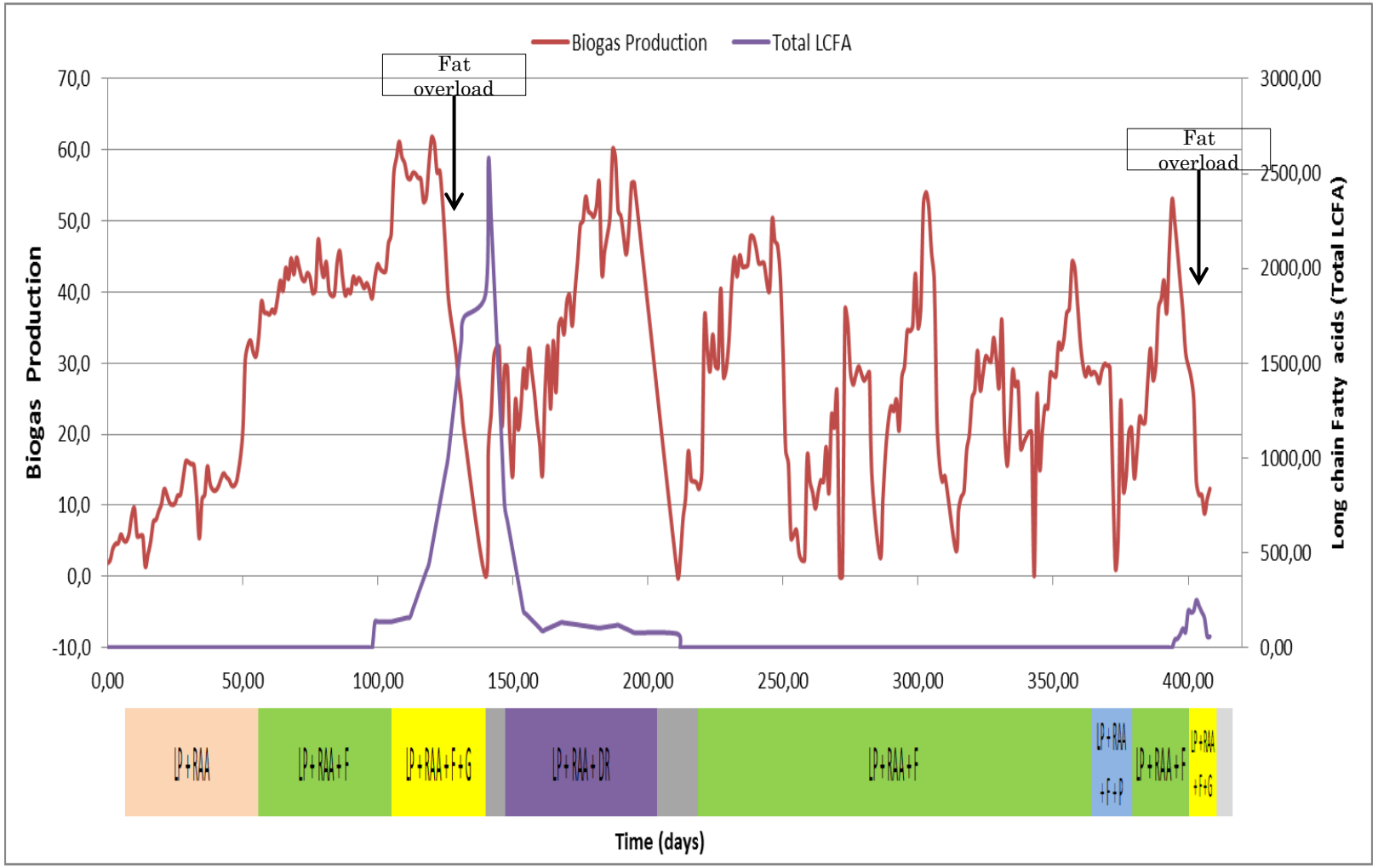
RESULTS DISCUSSION



- Global stability of the digester
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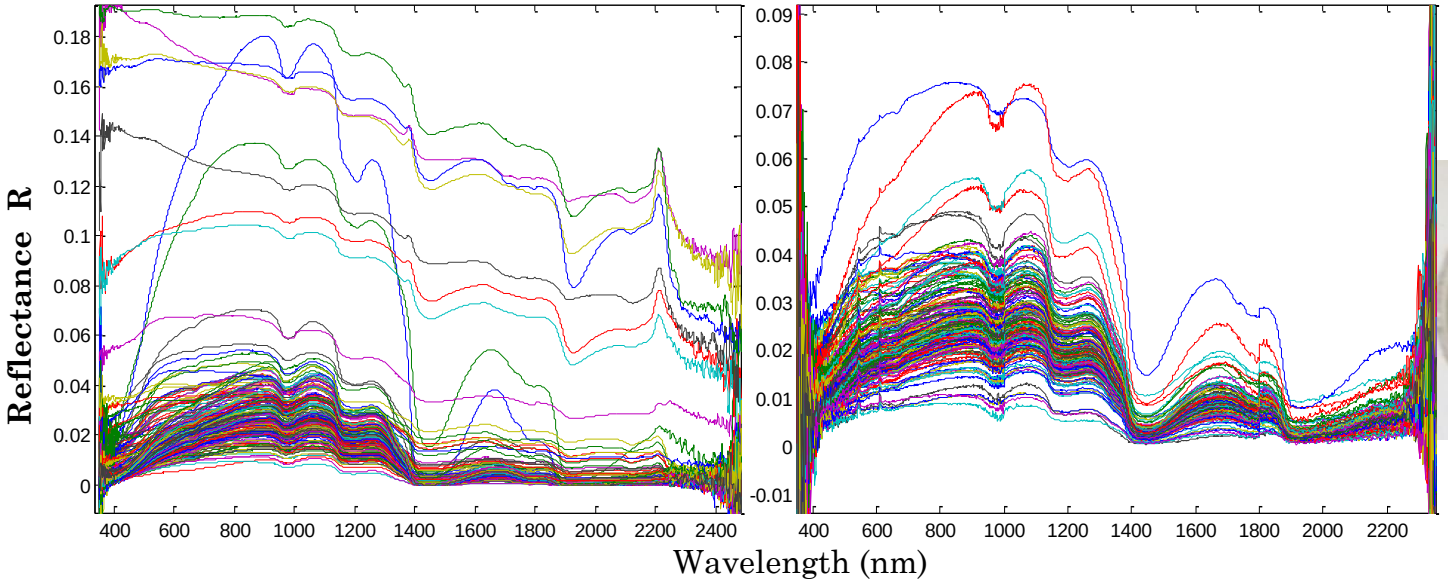
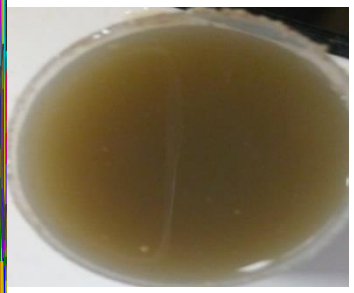
Results Discussion





TS: 13,6 – 26 g/kg. FM

2 fibers probe spectra

Polarized probe spectra (total scattering Rbs)

Impact of water: absorption dip at 970nm, 1200nm, 1440 nm and 1996 nm

- Logarithm
- Savitzky-Golay derivatives in order to evade parallel displacement
- Wavelength reduce to 450-2200nm

- Global stability of the digester
- NIRS Spectra and pretreatments
- PLS regression vs SO-PLS regression

Results Discussion

Descriptors block	Total VFA			Total LCFA			PCH4		
	LV	R2	SECV	LV	R2	SECV	LV	R2	SECV
2 fibers probe spectra X1(np)	12	0,58	2160	8	0,53	249	5	0,21	13,3
Polarized probe spectra X2(np)	11	0,61	2090	5	0,34	293	8	0,28	12,6
Chemical parameters X3(np)	6	0,91	974	6	0,27	308	6	0,47	10,8

55% 60% 13%

33% 66% 36%

Conclusion Perspectives

- AD process monitoring by NIRS on raw materials
- Water influence on the collected spectra
- Lack of robustness of mono-block predictions of VFA, LCFA and PCH_4
- Improvement of those predictions with the use of multi-block SO-PLS method
- Exploration of new multi-block model with polarized decomposition spectra

Analyse multi-bloc pour le suivi de la méthanisation

Thanks for your attention!

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