

## Multivariate process control charts based on reputed process indicators for the monitoring of anaerobic reactors

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### INTRODUCTION

So far various methods have been evaluated to monitor the anaerobic digestion (AD) process but none seems to be ideal. These methods usually consist in measuring a set of variables judged to be characteristic of the process status (i.e. pH of the liquid phase, CH<sub>4</sub>/CO<sub>2</sub> ratio of the biogas,...) and interpreting the collected data for each parameter individually. However, since these variables reflect the conditions of the reactor anaerobic microbial community, it appears very probable that they present a certain degree of correlation. An efficient tool for AD process monitoring should therefore benefit from the integration of information about the way the measured parameters interact when the process is in control. A method to satisfy this condition is to monitor the reactors using multivariate statistical process control (MSPC) as an alternative to usual univariate approaches. Several studies have been conducted to adapt MSPC techniques to AD process monitoring ([2],[1]). Nevertheless, these approaches use arbitrary measurable quantities as initial variables using e.g. near infrared spectroscopy or electronic nose, but no attempt has been done to exploit MSPC on the basis of initial variables commonly recognized as AD process stability indicators.

**AIMS OF THE STUDY:** (1) assessing the potential of Hotelling's T<sup>2</sup> charts built from individual parameters commonly recognized as process status indicators to interpretate the behaviour of a lab-scale continuously stirred anaerobic reactor (CSTR) progressively driven to an intoxication due to volatile fatty acid accumulation (i.e. acidosis); (2) evaluating if this multivariate approach provide an added value compared to classical univariate monitoring methods (individual X-bar charts).

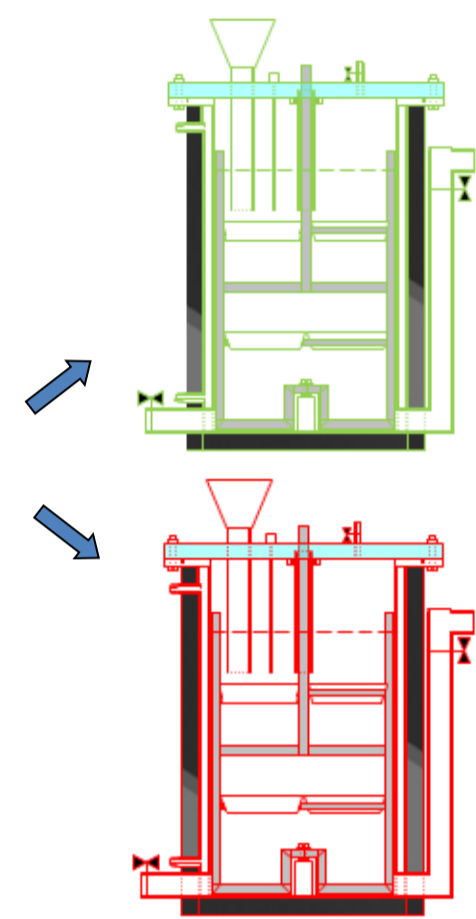
### MATERIAL & METHODS

#### Anaerobic digestion campaign targeting an acidosis

**2 CSTR**  
100l  
Mesophilic (37°C)

**Inoculum:** sludge from the anaerobic digester of a waste water treatment plant (mesophilic conditions).

**Substrate:** dried sugar beet pulps.



**1 steady state reactor (SSR):**  
Low organic loading rate (OLR) = 1,6 gVS.l<sup>-1</sup>.day<sup>-1</sup>  
Hydraulic retention time (HRT) = 28 days

**1 overfed reactor (OR):**  
OLR increased weekly by increments of 0,8 gVS.l<sup>-1</sup>.day<sup>-1</sup>  
HRT = 28 days

NB: The reactors were fed only during working days

#### Continuous measurement of individual parameters

**Gaseous phase:** Biogas production, [CH<sub>4</sub>], [CO<sub>2</sub>], [H<sub>2</sub>] - Hourly measurement frequency.

**Liquid phase:** Total solids (TS), Volatile solids (VS), total inorganic carbon (TIC), total ammoniac nitrogen (TAN) - Weekly measurement frequency.

**Process stability indicators:**  
pH, CH<sub>4</sub> yield, VFA<sub>sludge</sub>

#### Process monitoring using univariate and multivariate control charts (UVCC & MVCC)

Phase I = Model building for in-control process ;  
Phase II = Process monitoring using the model.

Phase I: SSR ; Phase II: OR

**UVCC:** Center line (CL): Average of the variable for phase I; Lower and upper control limits (LCL & UCL) : CL ± 2,68Stdev<sub>Phase I</sub><sup>1</sup>

#### MVCC:

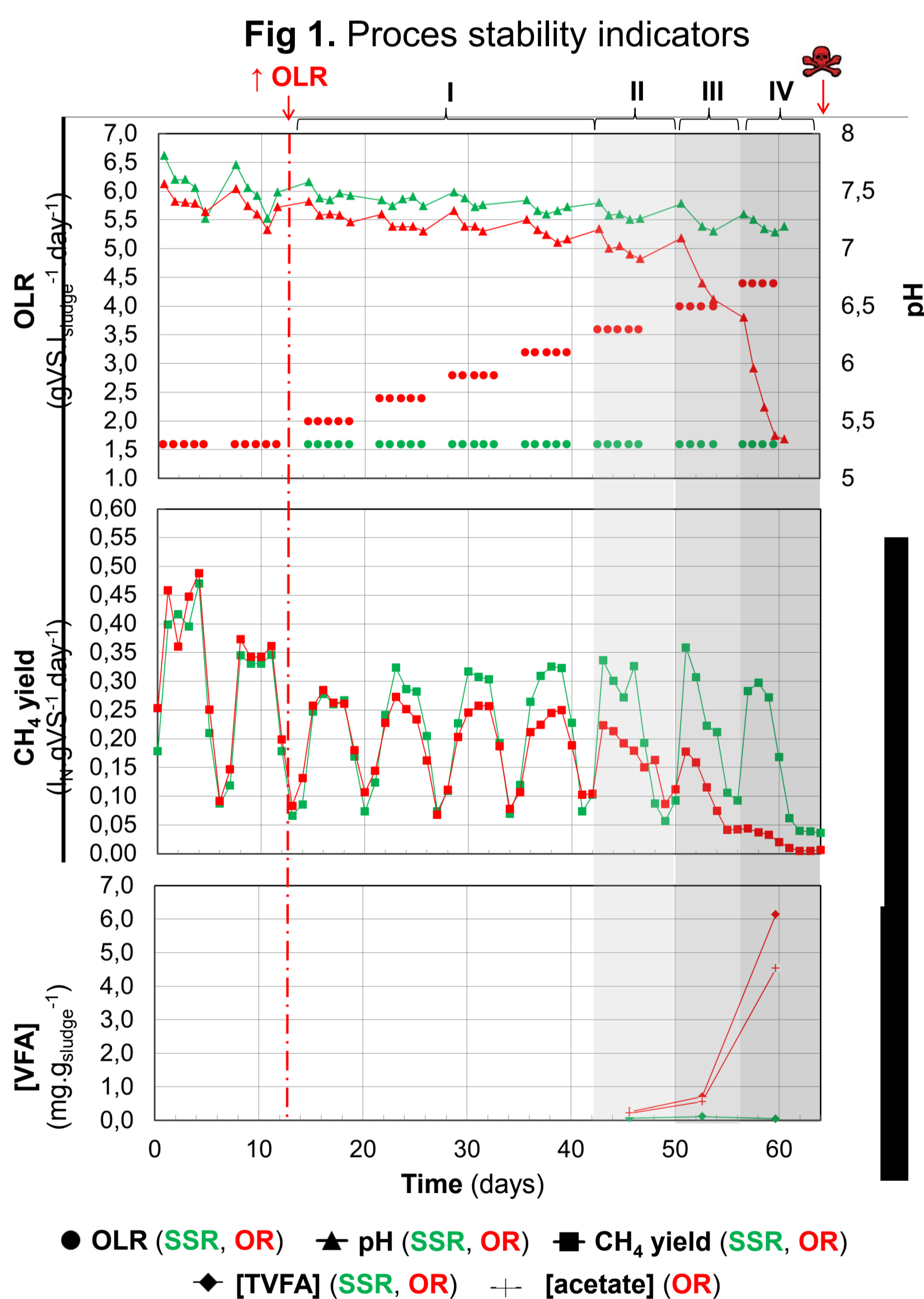
- Principal components analysis (PCA) → Hotelling's T<sup>2</sup> control chart computed on component score basis
- Components retained according to eigenvalue-one rule
- UCL for phase I and II calculated according to [3]<sup>1</sup>

<sup>1</sup>Risk of false alarm (α) = 0,01

### RESULTS & DISCUSSION

#### 1) Acidosis?

A critical VFA intoxication was reached at day 62 for the OR (Fig 1): No more biogas production, pH < 5,5, TVFA > 6mg/g<sub>sludge</sub>. For the SSR, the process indicators showed no sign of perturbation during the 65 days of the experiment.



● OLR (SSR, OR) ★ pH (SSR, OR) ■ CH<sub>4</sub> yield (SSR, OR)  
◆ [TVFA] (SSR, OR) + [acetate] (OR)

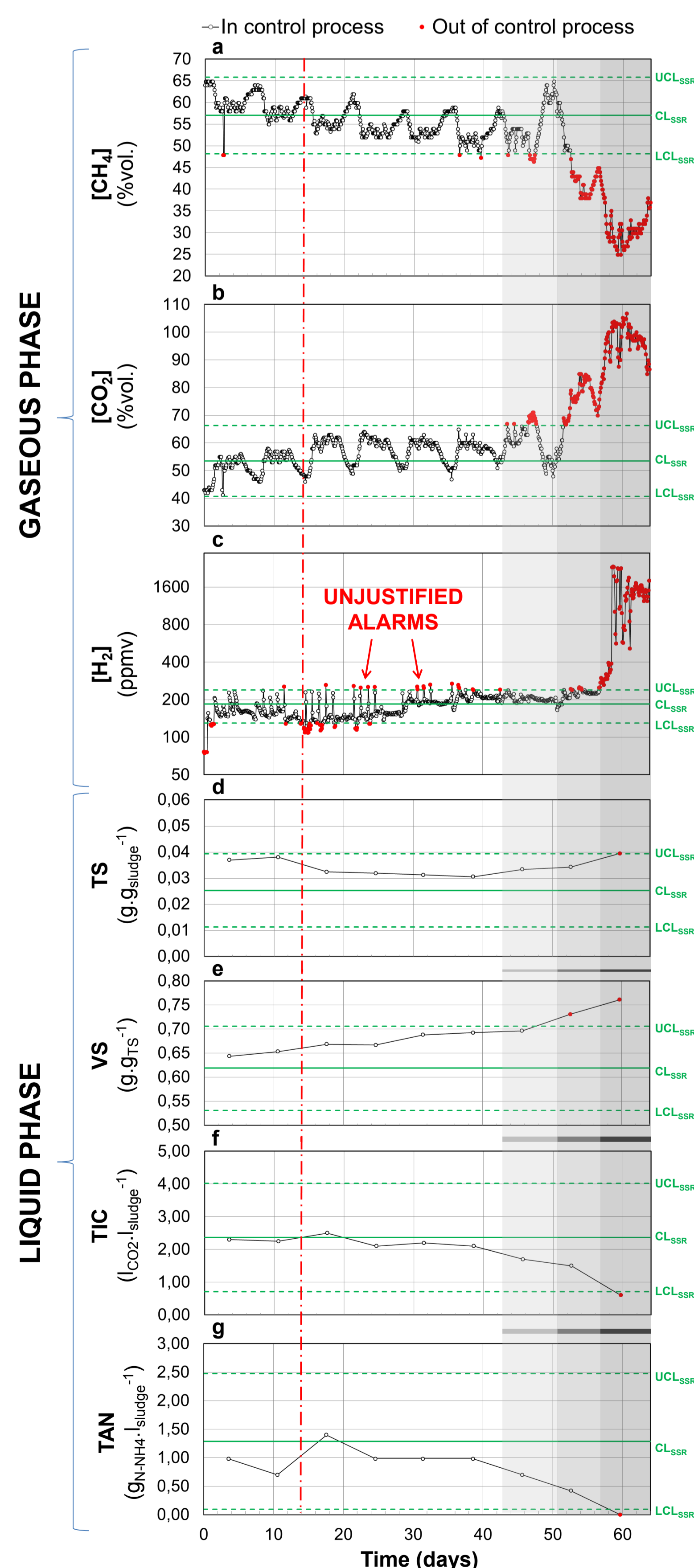
Table 1. 4 periods can be defined regarding OR performance from steady to collapsing state.

Period	OR process stability	Indicators
I	Stable	pH > 7 CH <sub>4</sub> yield stable during feeding days CO <sub>2</sub> content in biogas < 60%vol.
II	Affected	pH drop <sup>1</sup> : 7.2 → 6.8 CO <sub>2</sub> content in biogas → > 60%vol. <sup>1</sup> CH <sub>4</sub> yield ↓ during feeding days TVFA <sub>OR</sub> ~ TVFA <sub>SSR</sub>
III	Highly affected	pH drop <sup>2</sup> : 7.1 → 6.4 CH <sub>4</sub> yield ↓↓ during feeding days CO <sub>2</sub> content in biogas → > 70%vol. <sup>2</sup> TVFA > TVFA <sub>SSR</sub>
IV	Collapsing	pH drop <sup>2</sup> : 6.4 → 5.2 CH <sub>4</sub> yield ~ 0 TVFA <sub>OR</sub> >> TVFA <sub>SSR</sub> CO <sub>2</sub> content in biogas → ~ 80%vol. <sup>2</sup>

<sup>1</sup>Reversible after 2 days without feeding  
<sup>2</sup>Not reversible after 2 days without feeding

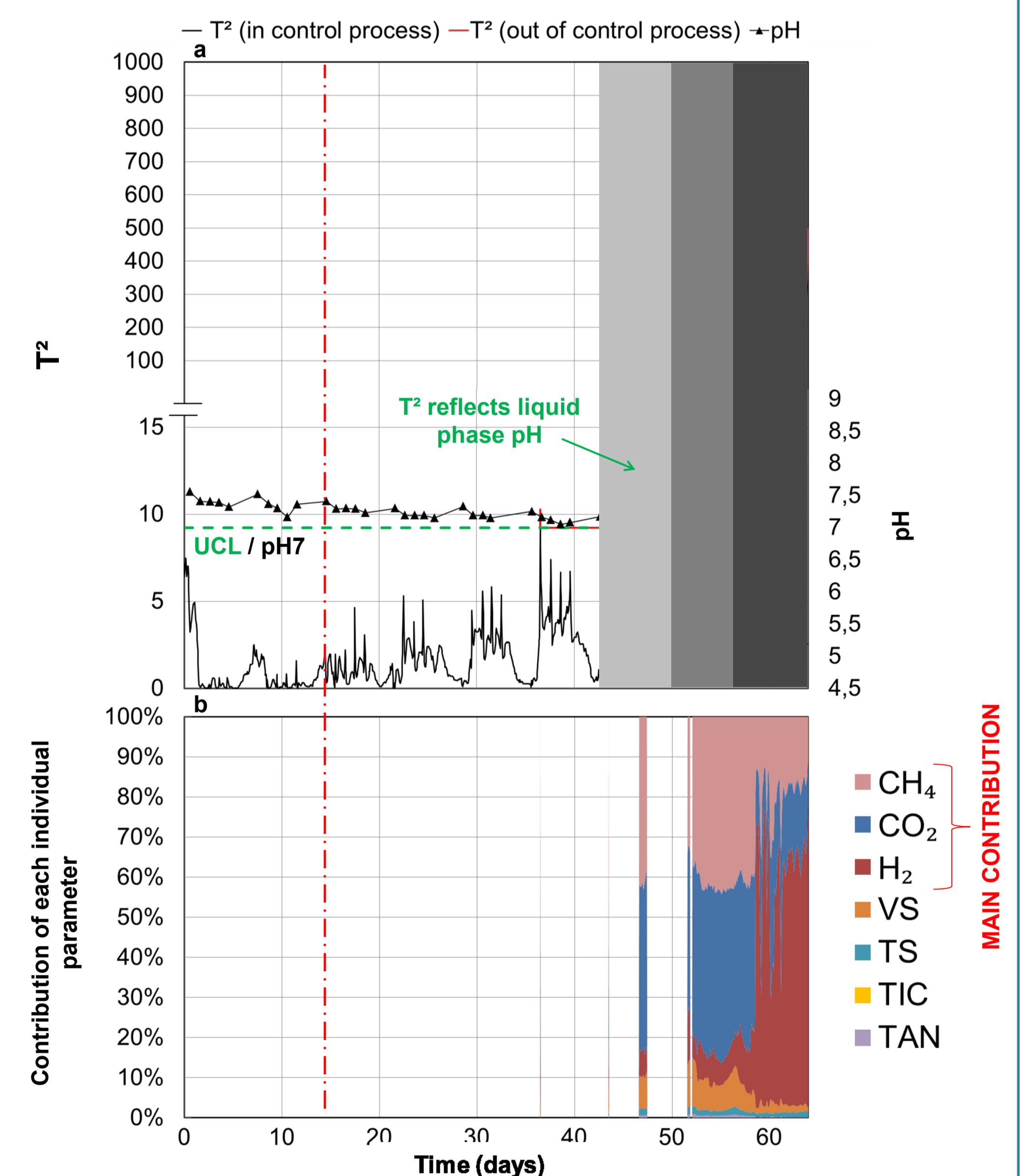
#### 2) Univariate monitoring

Fig 2. UVCC of each individual variable for the OR. LCL and UCL are computed on the basis of the SSR dataset.



#### 3) Multivariate monitoring

Fig 3. Hotelling's T<sup>2</sup> for the OR. Modelling parameters (covariance matrix & mean vector) are computed using the SSR dataset.



#### Highlights

- **Statistical process control** => promising tool to interpret the behavior of anaerobic reactor submitted to VFA intoxication. Exploiting an independent steady state reactor to define the in control baseline is possible.
- **Univariate control charts** detected unjustified out of control situations during stable process period (Fig. 2a, 2c). In addition, the reactor manager must integrate a large number of independent informations to judge process stability.
- The **multivariate approach** delivered a single signal easily comparable to its unique upper control limit. Approaching the acidosis, out of control T<sup>2</sup> values were in excellent accordance with the occurrence of acidic pH values.
- Hotelling's T<sup>2</sup> control charts built after PCA allow the easy construction of contribution charts => convenient diagnostic for each detected process perturbation (Fig. 3b). Gaseous phase parameters delivered the most valuable information about the process stability

#### References:

- [1] Adam, G., Lemaigre, S., Goux, X., Delfosse, P., & Romain, A. C. (2015). Upscaling of an electronic nose for completely stirred tank reactor stability monitoring from pilot-scale to real-scale agricultural co-digestion biogas plant. *Bioresource technology*, 178, 285-296.
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<http://www.optibiogaz.eu/>



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