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# How a two-day hydrolysis step improves the methane production rate in a TPAD reactor

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## Abstract

The influence of retention time and temperature on the hydrolytic stage of a high solids temperature phased anaerobic digestion system (TPAD) was investigated. The substrate was composed of cattle slurry and maize silage at different ratio (100, 70 and 30 % cattle slurry on VS basis) at constant total solids content of 12.8 %. It was incubated during 48h at 37, 55, 65 and 72 °C in order to simulate the hydrolytic stage of a TPAD. COD solubilisation and BMP were measured at 0, 24 and 48 h. The increase of temperature enhanced the amount of COD in the soluble phase. The influence of temperature on solubilization was thus in line with previously reported data, as higher temperatures brought fast higher COD turned into soluble form. Most of the solubilization took place in the first 24h of the hydrolytic stage. The most interesting result was that the kinetics of methane production in post-hydrolysis (evaluated by a BMP test) was greatly influenced by the hydrolysis time: no remarkable effect after 24h were noticed, but the kinetics of methane production was greatly enhanced after 48h (1<sup>st</sup> order kinetic coefficient increased by +86% on average). This effect was observed for the whole range of temperature tested. Surprisingly, no correlation was observed between the extent of solubilization and the improvement of the methane production kinetics. The behaviour of the particulate fraction of the sample was similar to that of the whole sample regarding this kinetics. Meanwhile, the intrinsic value of the BMP was not significantly modified by the hydrolysis stage.

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## Keywords

Temperature phased anaerobic reactor, hydrolysis; methane potential, methane production kinetics.

## INTRODUCTION

It is generally recognized that the limiting step of the anaerobic digestion of complex substrates is the hydrolytic step. This step may be biologically enhanced by a hydrolytic stage upstream the methane production reactor (Ge *et al.*, 2011). Since this stage can be optimized for temperature, the so-called combined hydrolytic + methane reactors were called temperature phased anaerobic digesters (TPAD). This technology has received a growing attention over the past 10 years. They are generally operated with a combination of a high temperature stage (for instance thermophilic) followed by a low temperature stage (for instance mesophilic). The high temperature stage is supposed to favour the hydrolysis rate of complex substrates, while the low temperature stage offers a stable operation for methane production (Sung & Santha, 2003). The first sector is thus dedicated to the hydrolysis at temperatures ranging from 55 °C to 65 °C or even 70 °C (Ge *et al.*, 2010). This first reactor is the most critical one in terms of performance. Positive effects of hydrolysis on BMP were observed by Orozco *et al.* (2013) for grass silage: at 4 days HRT for the hydrolytic reactor operated at 55°C, a 30% increase of the methane potential was observed in the subsequent methane production tests (BMP). In the present study, the effect of hydrolysis duration and temperature on hydrolysis performance and on subsequent methane production was investigated with a substrate composed of a mixture of cattle slurry and maize silage.

## MATERIAL AND METHODS

The feedstock used was composed of cattle slurry (12.8% TS, 71.0% VS/TS) and maize silage (26% TS, 96% VS/TS). Three different compositions were used: 100% cattle slurry (CS100), 70% cattle slurry (CS70) and 30% cattle slurry (CS30), expressed in amount of volatile solids. All the mixtures were homogenized and the TS content was adjusted at 12.8%. The hydrolytic reactor was a horizontal cylinder with a continuous mechanical stirring. It was insulated and heated with a thermal blanket. At the beginning of each experiment, the reactor was filled with 9 kg of feedstock input, purged with N<sub>2</sub> to ensure anaerobic conditions, and brought to the desired temperature. Once the desired temperature was reached, the initial sample (t<sub>0</sub>) was taken. Two other samples were taken at 24 and 48h. Samples were separated in two equal parts and processed as follows:

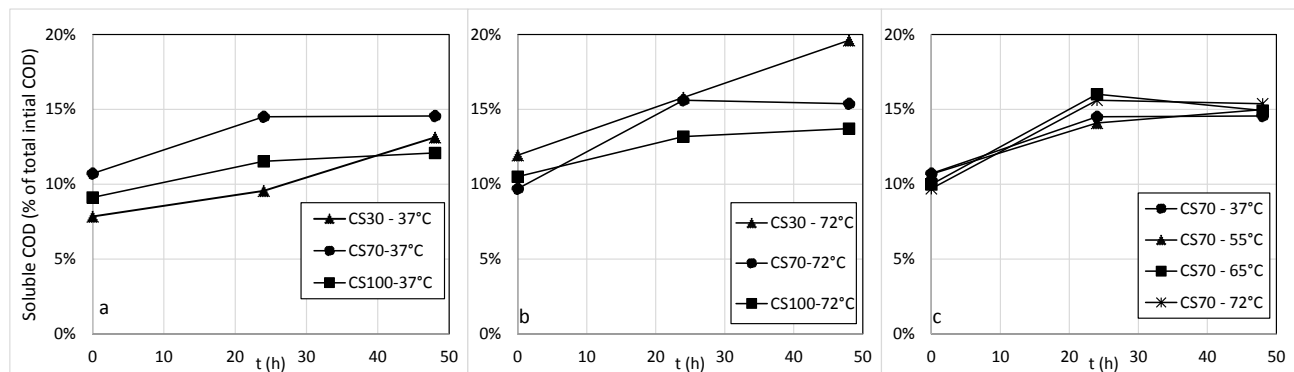
- Part 1 of the raw sample was analyzed for TS, VS and BMP;
- Part 2 was dedicated to liquid solid separation; water addition (to 10% TS), and centrifugation.
- Supernatant was filtered at 0.45 $\mu$  to obtain the soluble phase.
- Analysis on the soluble phase: COD (Hack kits), VFA (Gas Chromatography) and BMP.
- Analysis on the particulate phase (centrifugation bottom): TS, VS, BMP.

The BMP tests were performed according to the recommendation of the international dedicated task group on BMP harmonization (Holliger *et al.*, 2016). It was performed with the same inoculum for each run: this means that samples taken at t<sub>0</sub>, 24 and 48h underwent the BMP test with the same inoculum. BMP were done in triplicate for each subsample (raw sample, soluble part, centrifugation bottom) and for the blank.

## RESULTS AND DISCUSSION

The experimental conditions investigated were the following:

- Influence of substrate composition (CS30, CS70 and 100) at 37 and 72°C (3x2 = 6 runs);
- Influence of temperature at constant composition (CS70): 37, 55, 65 and 72°C (2 additional runs.)

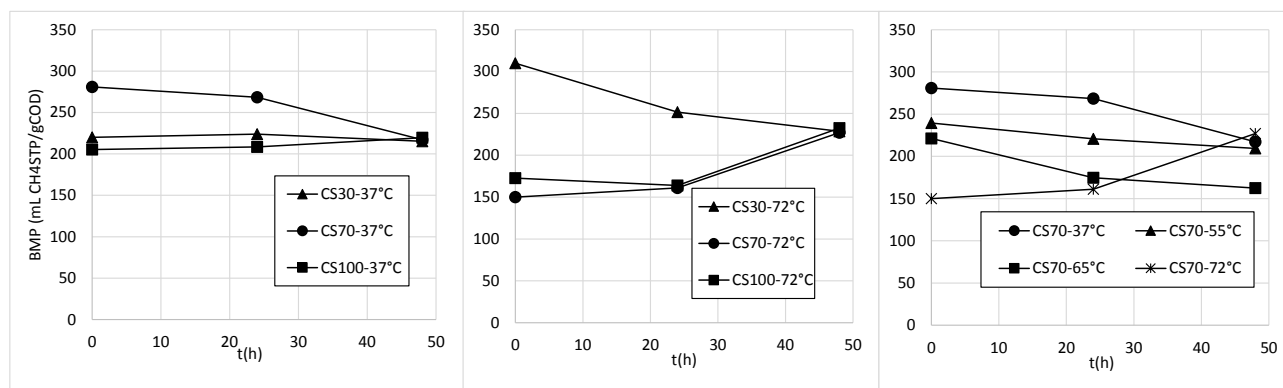


**Figure 1:** Extent of COD solubilization as a function of initial total COD. Influence of substrate composition at 37° (a), 72° (b); Influence of temperature at constant substrate composition (c).

### Hydrolysis and solubilization

The results for the solubilization are presented on Figure 1. For all tests, the initial soluble COD was comprised between 12 and 14 g/L (based on liquid content of the sample). COD solubilization occurred mostly within the first 24h, with exception for the substrate containing 30% slurry (CS30). Figure 1c shows that the effect of temperature after 24h was different at 37 / 55°C and at 65/72°C: at 37 and 55, the extent of solubilization moved from 10.7 to 14.5% (+35%) while it moved from 9.5 to more than 15% (gain +58%) at 65 and 72°C. Nevertheless, this difference was not confirmed after 48h hydrolysis. The contribution of the total VFA to the soluble COD was more important at 37°C

(80-85%) than at 72°C (50%), indicating that some hydrolysed matter were produced without acidogenesis at higher temperatures (results not shown in this abstract).

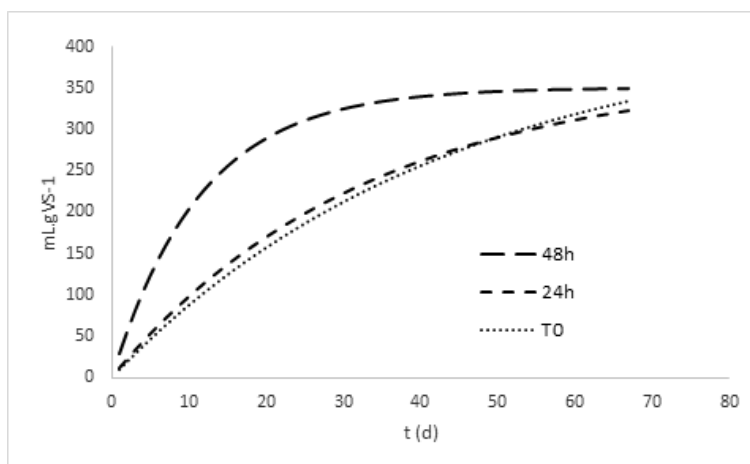


**Figure 2:** Evolution of the BMP during hydrolysis (data on raw samples).

### BMP and methane production rate

The BMP measurements did not evidence any trend of increase or decrease upon temperature, duration or initial composition. The only variation that was statistically significant was the increase of the BMP observed at 72°C after 48h for the 2 substrates containing maize silage (Figure 2, middle), but this effect of temperature was not confirmed by the data obtained for 100% cattle slurry at 72° and 70% - 65°C. The average contribution of the soluble part to the total methane production increased considerably with time: 11% at  $t_0$ , 14% at 24h and 18% at 48h. This contribution increased less for the tests performed at 72°C (10, 12 and 14% at  $t_0$ , 24h and 48h respectively).

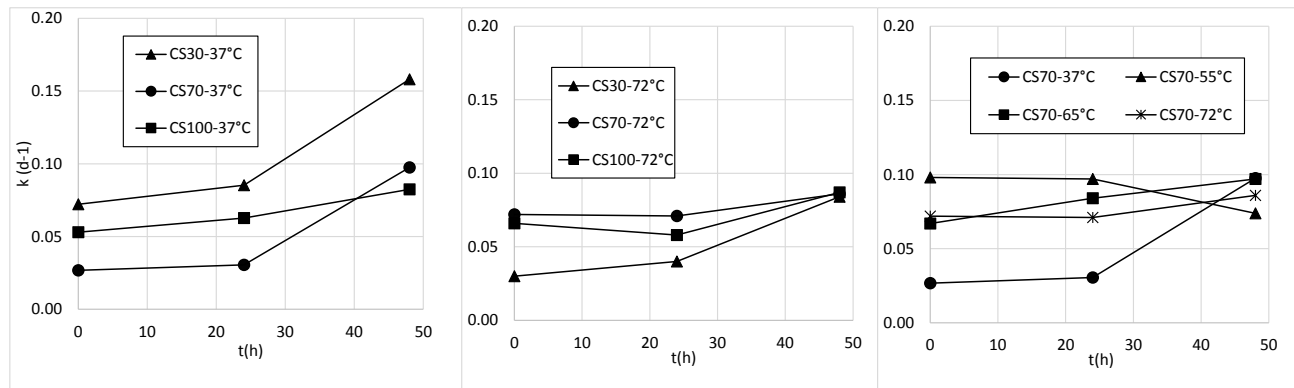
The most striking result observed concerned the evolution of the kinetics of methane production during the BMP tests following hydrolysis. Since these tests were done in parallel with the same inoculum for a given experimental condition, it is possible to compare the kinetics between BMP samples taken at  $t_0$ , 24h and 48h. A typical comparison of the methane production kinetics is presented on Figure 3.



**Figure 3:** Methane production (in mL STP /gVS) vs time during the BMP tests performed with raw sample CS70, 37°C (hydrolysis at 37°C, composition 70% cattle slurry).

The net methane production (net means that the methane production from the blank was retrieved) exhibit a first-order kinetics for all the tests. In the case presented on Fig 3, the first-order constant  $k$  was quite similar at 0 and 24h hydrolysis, but remarkably increased after 48h (0.027, 0.031 and 0.098  $d^{-1}$  respectively). The calculated 1<sup>st</sup> order constant are plotted on Figure 4. On average, the kinetics was multiplied by 1.12 after 24h and by 1.86 after 48h. The most striking increase was obtained at 37°C (x 2.5 after 48h hydrolysis). Hydrolysis temperature did not affect the increase of the methane

production kinetics. Only the test at 55°C did not follow this trend: during this test however, a significant production of biogas was observed during the 2-days hydrolysis. In addition, it can be seen from Figure 1 and 2 that both the soluble COD and the BMP decreased at 48h for the 55°C test, indicating probably that methane production has already occurred during hydrolysis.



**Figure 4:** 1<sup>st</sup> order constant of methane production during BMP as a function of pre-hydrolysis time.

## CONCLUSIONS

- The influence of temperature, time and substrate composition on hydrolytic performances were investigated in view of optimizing the first stage of a high-solid temperature-phased (TPAD) reactor treating cattle slurry and maize silage as co-substrates.
- Solubilization occurred in the first 24h of the hydrolysis step and was favored at higher temperature though at a limited extent.
- Downstream methane produced (estimated by BMP tests) was not significantly affected by the hydrolysis.
- Downstream methane production kinetics was greatly enhanced by the hydrolytic step and the 1<sup>st</sup> order constant for methane production increased by a factor 1.86 on average (max 2.5) after 48h hydrolysis.

## ACKNOWLEDGEMENTS

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