

Improving methane production during anaerobic digestion of the aqueous phase of hydrothermal carbonization: effect of intermediate treatment and choice of inoculum

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Anaerobic digestion (AD) has been identified several times as a promising process to deal with the aqueous phase produced during the hydrothermal carbonization (HTC) of organic waste (Campbell et al., 2022; Mannarino et al., 2022). In addition to reducing the organic concentration of this aqueous phase, AD can produce additional energy in the form of methane. Depending on the conditions, methane production can represent a non-negligible source of energy. It was demonstrated previously that in order to maximize the global energy yield of the integrated HTC-AD process, low temperatures should be used, since the methane yield decreases for aqueous phase produced at high temperatures (Brown et al., 2022; Yuan et al., 2019). However, improving the methane yield during the AD of the aqueous phase produced during HTC at high temperature could potentially improve the global energy yield and improve the hydrochar quality at the same time. The production of humic-like substances through Maillard reactions during HTC of organic waste is often reported as the main source of inhibition for AD. In theory, removing or degrading these compounds could improve the AD step. Another approach would be to adapt the microorganisms consortia to facilitate the degradation of such compounds.

In this project, three different approaches were tested to improve the methane yield obtained during the AD. Different intermediate treatments (enzymatic, chemical, physical), an adapted sludge and four different types of inocula (Table 1) were tested. The aqueous phases were obtained from the HTC at 240 °C of food waste.

Table 1: Characteristics of the different inocula

	Type	Temperature	Substrate	SS (g kg ⁻¹)	VSS (g kg ⁻¹)
Inoculum 1	Granular	Mesophilic	Fruits and vegetables	84.4	74.7
Inoculum 2	Granular	Mesophilic	Whey	72.9	53.4
Inoculum 3	Granular	Mesophilic	Fruits and vegetables	99.5	71.7
Inoculum 4	Non-granular	Mesophilic	Starch	5.0	4.7

For the first series of experiments (Figure 1), five treatments and an adapted inoculum were tested to see if it could improve the methane production obtained from the aqueous phase. The different treatments included the addition of NaOH (3% wt), the treatment in an ultrasound bath for 3 h at 80 Hz, the UV treatment at 254 nm for 5 h, the oxidation with air for 3 h at 40 °C and the treatment with Laccase enzyme with 500 U L⁻¹ at pH 5 for 24 h. Finally, an inoculum adapted in a UASB digester with aqueous phase from HTC for 7 weeks was also used.

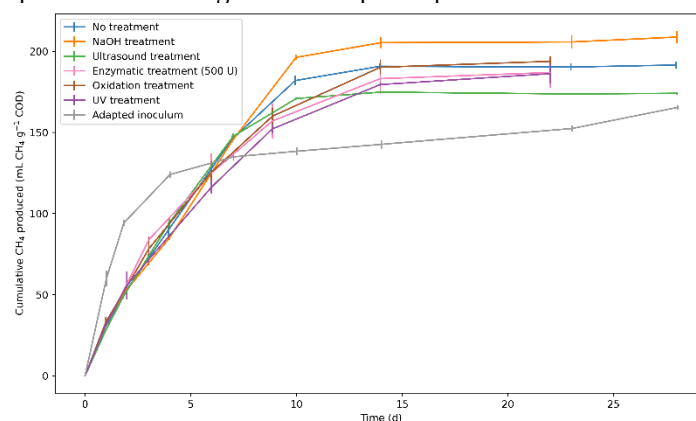


Figure 1: Effect of different treatments on the biochemical methane potential of the aqueous phase. Tests were carried out with inoculum 1. The aqueous phase was produced by the HTC of food waste at 240°C.

Results from this first phase showed no clear improvements of the conversion for all the treatment methods, except for the NaOH (improvement of 9%, p-value < 0.05). The oxidation, the enzyme and the UV treatments led to no significant impacts (p-value > 0.05), while the ultrasound treatment led to a decrease of performance (-9%, p-value > 0.05). Finally, the adapted inoculum led to a quicker production of methane, but a lower total production.

A second series of experiments (Figure 2) was carried out to investigate the effect of 3 of these treatments with more aggressive conditions. The NaOH treatment was carried out at 0.5 and 1 %, the enzyme treatment was

carried out at 2000 and 5000 U L⁻¹ and the UV treatment was carried out at 5 and 24 h. Finally, due to operational challenges of the industrial digester providing inoculum 1, this second series was done with a different one.

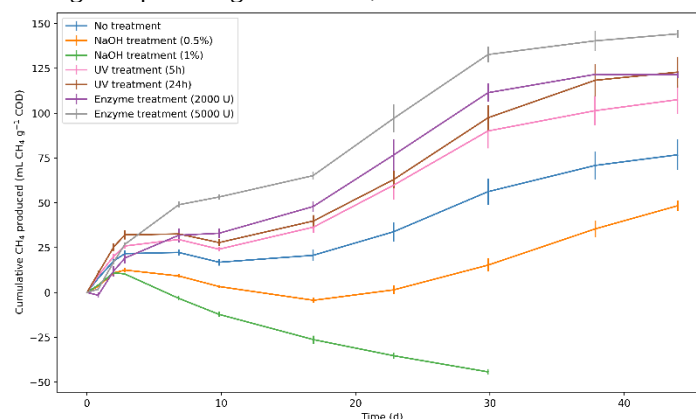


Figure 2: Effect of different treatments and conditions on the biochemical methane potential of the aqueous phase. Tests were carried out with inoculum 2. The aqueous phase was produced by the HTC of food waste at 240°C.

For this second series of experiments, it is clear that the second inoculum did not perform as well as the first one. An important lag phase was observed for the control (no treatment) and a much lower methane production was achieved. However, this second inoculum had similar activity test results (glucose, H₂, acetate and propionate) than the first one, indicating that the lower performance is not due to a lower activity of the inoculum. Additionally, it can be seen that the higher concentration of NaOH had the opposite effect by reducing considerably the methane production. However, both the UV treatment and the enzyme treatment showed promising results (statistically different for each case). The best improvement was obtained for the enzyme treatment at 5000 U L⁻¹, the UV treatment of 24 h, the enzyme treatment at 2000 U L⁻¹ and the UV treatment of 2000 U L⁻¹, with increases of 87%, 60%, 58% and 40%, respectively.

Finally, since important differences were observed for the two tests, two additional tests (Figure 3) were carried out with different types of inocula. The characteristics of the inocula are presented in Table 1.

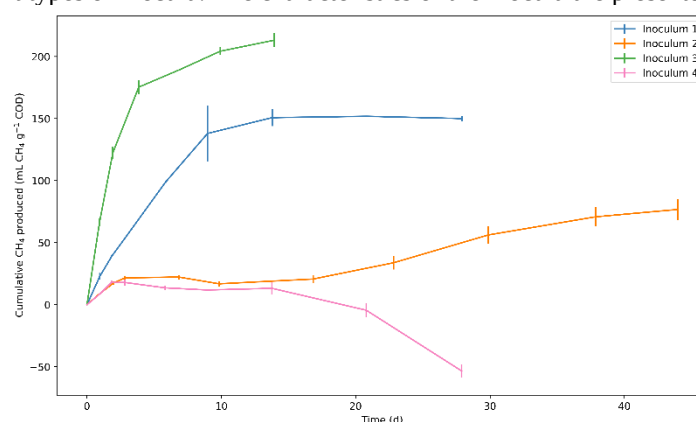


Figure 3: Effect of different inocula on the biochemical methane potential of the aqueous phase. The aqueous phase was produced by the HTC of food waste at 240°C.

It can clearly be seen that the inoculum has an important effect on the methane production from the aqueous phase. The inoculum obtained from a starch thermophilic digester was strongly inhibited, as well as for the whey mesophilic inoculum. Only the inocula from fruit and vegetables digesters performed adequately.

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