

# Pre-treatment of sewage sludge to enhance methane production: optimal technology's evaluation through continuous reactor operation.

Mitraka G.C.<sup>1</sup>, Kontogiannopoulos K.N.<sup>1</sup>, Zouboulis A.I.<sup>2</sup>, Kougias P.G.<sup>1</sup>

<sup>1</sup>Soil and Water Resources Institute, Hellenic Agricultural Organization – Dimitra, Themi, 57001, Greece

<sup>2</sup>Laboratory of Chemical Technology, Department of Chemistry, Aristotle University of Thessaloniki, Thessaloniki, 54124, Greece

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Presenting author e-mail: c.mitraka@swri.gr

Considering that world is on an unsustainable path and significant inequalities in energy consumption and access to energy persist, the need to develop new strategies to address these issues is urgent. On those accounts, “Waste to Energy” technologies are emerging since they offer an attractive alternative to fossil-based fuels. The large quantities of sewage sludge produced worldwide by municipal wastewater treatment plants (WWTPs) render this substrate as an attractive second-generation biofuel feedstock, that has imposed a global challenge for its valorization and management (Khanh Nguyen et al., 2021). Thus, sewage sludge is classified among the various organic wastes being anaerobically digested with concomitant production of biogas. However, this substrate's complex composition of refractory products and extracellular polymeric substances (EPS) withstands biological conversion through anaerobic digestion process, leading to the need of pre-treatment technologies to increase its biodegradability (Sakaveli et al., 2021).

The present study was undertaken to evaluate fourteen (14) different methods for sewage sludge pre-treatment and identify the optimal one so ensuring increased degradation of biomass and facilitate the access of microorganisms and enzymes to the biodegradable organic matter contained within the sewage sludge. The efficiency of each pre-treatment technology was examined through proper comparisons in terms of methane productivity between the pre-treated samples at various conditions, which was evaluated by Biochemical Methane Potential (BMP) assays. Finally, the most promising pre-treatment method is further evaluated in continuous reactor operation.

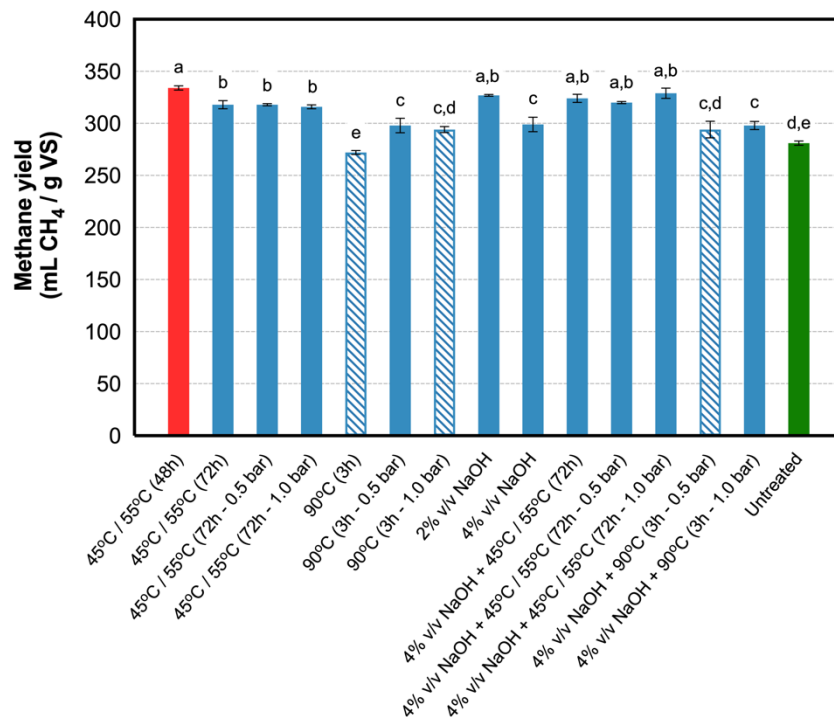
All the pre-treatments were performed on a mixture of primary and secondary sludge (PSS) collected from the Thessaloniki's Municipal Wastewater Treatment Plant (MWWTP). As depicted in Table 1, the examined pre-treatment methods, include thermal and alkaline hydrolysis. Nonetheless, since the simultaneous application of these techniques has been considered as sufficient for the disintegration of sludge compared to when they are applied individually (Ariunbaatar et al., 2014, Atelge et al., 2020), their combination was also assessed. The aim was to evaluate whether combined pre-treatment can further improve the biodegradability of the substrate and lead to an even higher methane production during AD. Additionally, some of the pre-treatments were conducted under 0.5 and 1.0 bar of pressure achieved by using carbon dioxide (CO<sub>2</sub>). Both chemical and thermochemical pre-treatments were carried out using NaOH 1 M, a frequently applied alkaline reagent with significant disintegration efficacy (Shehu et al., 2012, Kim et al., 2013). Each experiment (treatment) was carried out in triplicate.

During BMP testing which was set according to the methodology described in the BMP protocol (Angelidaki et al., 2009), the amount of produced methane was measured regularly using gas chromatography until no further methane was generated from each treatment. Given that the untreated sample produced 280 mL CH<sub>4</sub>/g VS, it was found that all the applied pre-treatment methods, contributed to an increment in the methane yield in a percentage ranging from 5 to 19%. Statistical analysis was applied to assess whether these values presented a statistically significant difference compared to the untreated trial (Figure 1). The results revealed that eight (8) out of the fourteen (14) examined pre-treatment methods delivered a *p*-value (*p*\*) less than 0.05, indicating the statistical significance of their differences compared to the untreated trial. Nonetheless, for the samples treated with 2% v/v and 4% v/v NaOH, non-statistically significant differences were detected. The same applied for the ones undergone alkaline hydrolysis in combination with a two-phase thermal pretreatment at 45° C and 55° C for 72 h respectively, as well as for the samples treated at the same conditions with an additional 0.5 and 1.0 bar of pressure. Along with the abovementioned samples, the difference of those treated at 90 ° C for 3 h under 0.5 bar was statistically insignificant, presenting a *p*-value > 0.05.

**Table 1.** Conditions applied for sewage sludge pre-treatment.

Thermal Hydrolysis	
1	45° C for 48 h and then 55° C for extra 48 h
2	45° C for 72 h and then 55° C for extra 72 h
3	45° C for 72 h and then 55° C for extra 72 h under 0.5 bar of CO <sub>2</sub> pressure
4	45° C for 72 h and then 55° C for extra 72 h under 1.0 bar of CO <sub>2</sub> pressure
5	90° C for 3 h
6	90° C for 3 h under 0.5 bar of CO <sub>2</sub> pressure
7	90° C for 3 h under 1 bar of CO <sub>2</sub> pressure
Alkaline Hydrolysis	
8	NaOH 2% v/v
9	NaOH 4% v/v
Thermochemical Pre-treatment	
10	NaOH 4% v/v, 45° C for 72 h and then 55° C for extra 72 h
11	NaOH 4% v/v, 45° C for 72 h and then 55° C for extra 72 h, under 0.5 bar of CO <sub>2</sub> pressure
12	NaOH 4% v/v, 45° C for 72 h and then 55° C for extra 72 h, under 1 bar of CO <sub>2</sub> pressure
13	NaOH 4% v/v and 90° C for 3 h, under 0.5 bar of CO <sub>2</sub> pressure
14	NaOH 4% v/v and 90° C for 3 h, under 1 bar of CO <sub>2</sub> pressure

The pre-treatment which appeared to be the most efficient, presenting an increment of 19% in methane yield, was conducted in two phases, during which the samples were first treated at 45° C for 48 h and then transferred to a preheated incubator at 55° C, where they remained for additional 48 h. The obtained results are in line with those reported by a previous study concerning sludge pretreatment under corresponding operating parameters. Particularly, Ding et al. (2017) concluded that the hydrolysis rate increases with increasing temperature from 35° C to 55° C and that biological hydrolysis methods at 42° C to 55° C achieve a statistically significant increase in the amount of methane produced, compared to the amount produced by anaerobic degradation of untreated sludge. These observations are ascribable to the fact that low-thermal pre-treatment aids in hydrolyzing complex organic constituents of sludge through increasing the activity of thermophilic bacteria that promote the release of hydrolytic enzymes in the substrate. In addition, this pre-treatment causes sludge solubilization, and thus, increases the amount of utilizable organic matter (Neumann et al., 2016, Taşeli, 2020).



**Figure 1.** Bar chart of statistically significant differences between the pre-treated samples, regarding methane yield. Different letters above the bars signify distinct statistical groups ( $p < 0.05$ ) between the applied pretreatment methods.

In current phase, a set-up of three replicate continuous stirred reactors (CSTR) was settled with a total and working volume of 2.0 L and 1.5 L, respectively. The reactors operate under mesophilic (37° C) conditions and PSS treated according to the method selected (45° C for 48 h and 55° C for additional 48 h) is used as feedstock. The organic loading rate (OLR) of the reactors is 2 gVS L<sup>-1</sup> d<sup>-1</sup>. At present, the experiment is ongoing and results from the continuous reactor operation will be available upon the conference time.

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