Trade-offs between hydrochar quality and liquid fraction biodegradability in sewage sludge hydrothermal carbonization

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1. Introduction

The management of sewage sludge (SS) remains a major environmental and operational challenge for wastewater treatment plants (WWTP), due to increasing sludge generation, stricter environmental regulations and the need to improve resource recovery within a circular economy framework. Conventional disposal routes, such as landfilling, incineration or direct land application, are progressively limited and often fail to exploit the energetic and material potential of this residue. Hydrothermal carbonization (HTC) has emerged as a promising thermochemical technology for the treatment of wet biomass, particularly sewage sludge, as it operates in an aqueous environment at moderate temperatures (180–300 °C) and autogenous pressure, avoiding the need for prior drying (Hidalgo et al., 2024). HTC produces three main fractions: a solid hydrochar, a liquid fraction (LF) rich in solubilized organic compounds, and a gaseous fraction mainly composed of CO_2. In most HTC studies, process optimization has traditionally focused on maximizing gas production or improving hydrochar yield and quality, while the liquid fraction has frequently been treated as a secondary by-product or even as a residual stream requiring disposal. As a result, the potential role of the liquid fraction in integrated energy recovery schemes has often been overlooked. In parallel, several strategies have been proposed to enhance hydrochar properties (such as modifying HTC operating conditions or altering the feedstock composition) to increase its carbon content and energetic value. Among them, the co-hydrothermal carbonization of sewage sludge with additional carbon-rich substrates, such as pyrolysis-derived biooil (BO), has been shown to significantly improve hydrochar characteristics (Petrovič et al., 2024). However, changes in feedstock composition and process configuration not only affect the solid product but also strongly modify the composition of the resulting liquid fraction, with direct implications for its biodegradability and downstream valorization. Previous studies (Hidalgo et al., 2025) have shown that HTC operating conditions strongly influence both hydrochar quality and the composition and biodegradability of the liquid fraction. Therefore, a comprehensive assessment of HTC performance requires an integrated evaluation of all generated fractions, particularly when feedstock modification strategies are applied. In this context, the present work focuses on the biodegradability and biomethane potential of the liquid fraction generated during sewage sludge HTC, both when sewage sludge is treated alone and when co-hydrothermal carbonization with pyrolysis-derived biooil is applied. Building on a previously selected mild HTC baseline (180 °C, 60 min, pH 3), the study evaluates (i) the robustness of anaerobic digestion under increased liquid-fraction dosages and different co-substrate scenarios, and (ii) how feedstock modification through co-carbonization alters the downstream anaerobic performance of the resulting aqueous stream.

2. Experimental conditions

HTC experiments were conducted using a laboratory-scale batch reactor with a working volume of 0.34 L (Figure 1a). A mild baseline HTC condition (180 °C, 60 min, initial pH 3) was selected as reference to generate the liquid fraction (LF) for biodegradability assessment. In parallel, co-hydrothermal carbonization trials were performed under the same baseline conditions using sewage sludge (SS) blended with pyrolysis-derived bio-oil (BO) at different SS:BO ratios (Table 1), in order to investigate how feedstock modification affects the anaerobic performance of the associated liquid stream. Anaerobic biodegradability tests (BMP) were carried out in 1 L stainless steel reactors with a working volume of 500 mL (Figure 1b) under mesophilic conditions (34 ± 1 °C) and constant agitation. The inoculum consisted of anaerobic sludge (digestate) collected from a full-scale municipal WWTP (14.7 $gVS\cdot L^{-1}$), pre-incubated for 72 h at 35 °C to reduce residual biodegradability. The LF obtained from baseline HTC of SS presented 10.7 $gVS\cdot L^{-1}$. Biodegradability assays were conducted under co-digestion conditions using pig manure as co-substrate (3.9 $gVS\cdot L^{-1}$), selected as a broadly representative substrate in anaerobic digestion systems. Three experimental tiers were defined. In Tier 1, 5 mL of LF was added to pig manure and inoculum to achieve a substrate-to-inoculum ratio (S/X) of 0.5, enabling screening and comparability across LF samples. In Tier 2, the LF dose was increased to 50 mL per reactor to assess process robustness and potential inhibition at higher liquid-fraction loading. In Tier 3, LF samples obtained from co-HTC of SS and BO at different mixing ratios (Table 1) were evaluated under the same BMP approach (5 mL LF addition) to determine how co-feeding alters the biodegradability and biomethane potential of the resulting aqueous fraction. Biogas production was monitored via headspace pressure sensors, and gas composition was measured using a Varian CP-4900 Micro-GC equipped with thermal conductivity detectors.

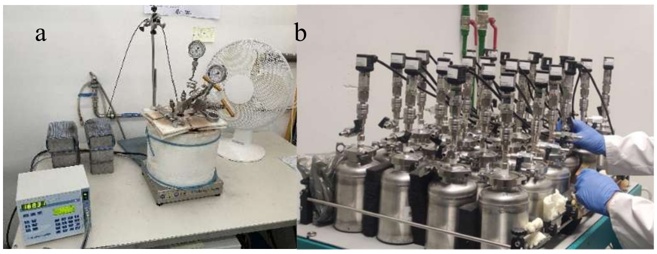

Figure 1: a) HTC lab-scale reactor; b) Anaerobic digestion test.

Table 1. Experimental conditions.

	HTC process (190º C/1h/pH 3)		BMP process	
	Feedstock	%	LF addition	Co-substrate
Tier 1	SS	100	5 mL	Pig manure
Tier 2	SS	100	50 mL	
Tier 3	SS: BO (=Tier 1)	100:0	5 mL	
	SS: BO	75:25	5 mL	
	SS: BO	50:50	5 mL	
	SS: BO	25:75	5 mL	
	SS: BO	0:100	5 mL	

SS: Sewage Sludge; BO: Biooil; LF: Liquid fraction from HTC

3. Results

Figure 3 summarizes methane production across the three experimental tiers. In Tier 1, the addition of the LF obtained from HTC of SS increased methane production compared to the blank (pig manure + inoculum), confirming that HTC process water is biodegradable and can act as an effective co-substrate. In Tier 2, increasing the LF dose (50 mL per 500 mL reactor) did not show signs of inhibition and resulted in higher cumulative methane production at reactor scale, supporting the robustness of co-digestion under higher LF loading. In Tier 3, co-hydrothermal carbonization of SS with pyrolysis-derived BO led to a clear negative trend: methane production decreased progressively as the BO share in the initial SS:BO mixture increased, indicating that feedstock modification aimed at solid upgrading can adversely affect the downstream anaerobic performance of the associated liquid stream.

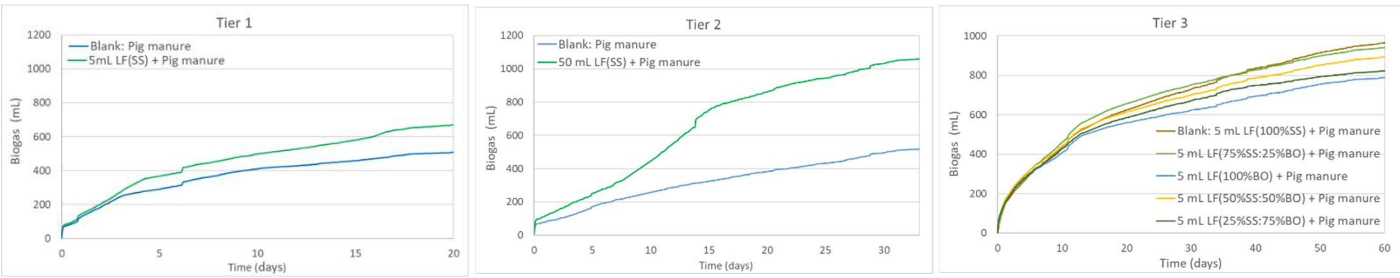

Figure 2: Experimental results.

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