

### THERMAL HYDROLYSIS PRE-TREATMENT FOR MECHANICALLY SORTED ORGANIC WASTE. ANAEROBIC CO-DIGESTION WITH SEWAGE SLUDGE

### **Highlights**

- Thermal hydrolysis enhances substrates bioavaibility and improved anaerobic digestion performance.
- Organic matter removal is up to 35% higher with co-digestion than with mono-digestion.
- Co-digestion increases methane production up to 14% compared to mixed sewage sludge.

#### Introduction

Municipal solid waste (MSW) management is an increasing global challenge due to population growth and urbanization. To address this, sustainable waste handling efforts have reduced landfill disposal and promoted recycling. The European Union aims to recycle 65% of MSW and limit landfill use to 10% by 2035. Mechanical biological treatments (MBT) play a key role in handling unsorted MSW. By 2016, around 570 MBT plants operated in Europe, with future expansion expected (Cesaro and Belgiorno, 2021). MBT recovers materials by separating metals for recycling, extracting combustible fractions for energy recovery, and stabilizing organic residues.

Anaerobic digestion (AD) is an alternative to aerobic stabilization for processing MBT-derived organic fractions. AD offers a sustainable solution by producing methane-rich biogas instead of consuming energy for aeration. Furthermore, anaerobic co-digestion (AcoD) presents effective ways to process organic waste, generating methane while reducing pollution. In addition, co-digestion enables the achievement of an optimal C:N ratio, within the ideal range of 20:1-30:1, thereby enhancing the biological efficiency (Bouallagui et al., 2009). However, impurities and toxic compounds in the organic fraction, as well as the slow hydrolysis of these low-bioavailability raw materials, hamper the efficiency of AD and AcoD, requiring better pre-treatment strategies to improve waste suitability. Various pre-treatment methods, including thermal, biological, chemical, and mechanical approaches, have been investigated to accelerate hydrolysis and improve process performance. Thermal hydrolysis (TH) is widely applied in full-scale AD plants for sewage sludge treatment, but its application to organic waste remains challenging.

Econward's Biomak® is a patented industrial thermal hydrolysis technology designed to enhance AD. This semi-continuous process homogenizes, degrades, and sanitizes solid organic waste, improving its suitability for AD while maintaining low energy consumption and achieving over 90% organic fraction recovery. Biomak® can process different waste types, including mixed municipal waste, source-separated organics, and food waste. This low-temperature operating technology (130 – 152 °C) facilitates inorganics separation to produce a cleaner substrate with higher methane potential.

The aim of this research is to evaluate the impact of the Biomak® thermal hydrolysis process on the co-digestion of hydrolyzed mechanically sorted organic waste with sewage sludge. For this purpose, sorted organic waste pretreated via thermal hydrolysis at 152  $^{\circ}$ C - 4 bar, and set-up to ensure influent requirements, was subjected to batch AcoD experiments containing between 100% and 70% volume of sewage sludge, to optimize methane production.

### **Material and Methods**

The anaerobic digestate, collected from a mesophilic reactor of a municipal wastewater treatment plant (Madrid, Spain), was used as inoculum to carry out the experiments. Its main characteristics were total solids (TS), 32.1 g/kg; volatile solids (VS), 18.4 g/kg; and total chemical oxygen demand (TCOD), 36.9 g/L.

Mechanically sorted organic fraction (MOF) obtained through MBT, from MSW and collected from a waste treatment plant near Madrid, Spain, was used as raw material. Thermal hydrolysis of MOF was carried out in the Biomak® (Econward Tech, S.L.U) under 4 bar and 152 °C. Wet feedstock (1.3 t, 50% moisture content) was treated for 20 min. After hydrolysis, the biomass was screened through a 40 mm light sieve. The resulting biomass was subjected to cleaning using water in an industrial system consisting of a pulper, a hydrocyclone and a rejector, to



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obtain a cleansed and diluted material called biopulp. The main characteristics of the raw substrates, mixed sewage sludge (MSS)(composed by the mixture of concentrated primary and secondary sludges) and biopulp (B), are collected in **Table 1**. The MSS used as a control as well as the five mixtures used to carry out the batch AcoD experiments are presented in **Table 2**, named by the volume of biopulp (B) added (e.g., B-5, for a 5% volume of biopulp added).

**Table 1** Characterization of mixed sewage sludge and biopulp.

	MSS	Biopulp
TS(g/kg)	52.0(0.3)	50.6 (0.9)
VS(g/kg)	40.7(0.2)	37.1(0.7)
TCOD(g/L)	66.5 (0.8)	68.9 (0.8)
SCOD(g/L)	3.6 (0.1)	18.9 (0.4)
VFA(gCOD/L)	2.3(0.0)	4.5 (0.0)
TOC(g/L)	0.9(0.0)	6.2 (0.3)
TKN (g/L)	2.2 (0.1)	1.3 (0.0)
$N-NH_3(g/L)$	0.1(0.0)	0.3(0.0)
рН	5.8(0.0)	5.5 (0.0)
Total alkalinity (g CaCO <sub>3</sub> /L)	1.2 (0.0)	2.2(0.0)

Standard deviation is reported in parenthesis.

**Table 2** Substrates used for batch experiments.

Mixtures	MSS	Biopulp
samples	(% volume)	(%volume)
B-0	100	0
B-5	95	5
B-10	90	10
B-15	85	15
B-20	80	20
B-30	70	30
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Triplicate batch AD and AcoD mesophilic runs ( $35\,^{\circ}$ C) were carried out in 120 mL glass vials, including a nutrient solution after which the vials were made up to volume ( $60\,\text{mL}$ ) with deionized water. The headspace of the vials was gassed with N<sub>2</sub> for 3 min to ensure anaerobic conditions. The initial inoculum concentration was set at 12 gVS/L and an inoculum-to-substrate ratio (ISR) of 1.1, on a VS basis. Moreover, duplicate tests for the blank runs (samples establishing the biogas level from the inoculum), and for the starch positive control runs (samples determining the inoculum activity) were used. Biogas volume and composition were measured nine times through the experiment, every day for the first three days of the trial, twice a week for the next two weeks, and weekly until the end of the experiment. At the same time as the biogas measurement (seven times) a vial of each mixture sample was sacrificed to monitor the evolution of the process. The biochemical methane potential (BMP) was calculated by subtracting the amount of methane produced by the blank experiments and relating it to the amount of TCOD. The trial was extended for 30 days.

TS, VS, soluble COD (SCOD), total Kjeldahl nitrogen (TKN), ammonia nitrogen (N-NH $_3$ ) and pH were determined by APHA methods (2005). TCOD was analyzed using the method proposed by Raposo et al. (2008). The concentrations of individual volatile fatty acids (VFA) from acetic to heptanoic, iso-forms included, were determined by gas chromatography (GC) on a ShimadzuGC-230 instrument equipped with a flame ionization detector (FID) (Diez et al., 2024). Total organic carbon (TOC) of the soluble fractions was measured using a TOC-VCPN analyzer (Colin et al., 2025). Total alkalinity was measured following the procedures recommended by Jenkins et al. (1983). Biogas production was assessed manometrically (Rozzi and Remigi, 2004) by measuring the pressure in each vial with a digital manometer (Sika) and expressed at standard temperature and pressure (STP: 273 K, 1 bar). Biogas composition (H $_2$ , N $_2$ , CH $_4$  and CO $_2$ ) was determined by GC separation on a Shimadzu GC-2014 unit equipped with a Carboxen 1010 PLOT fused silica capillary column and a thermal conductivity detector (TCD).

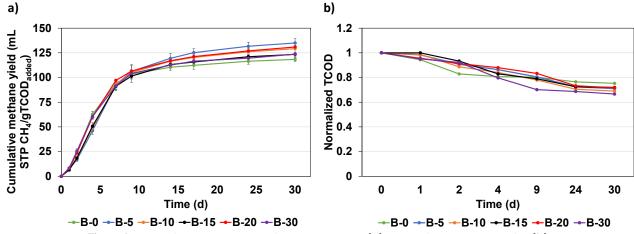
### **Results and Discussion**

Biopulp and MSS showed similar VS/TS ratios (73% and 78%). A key advantage of biopulp is its higher SCOD/TCOD ratio (27%) compared to MSS (5%). Biopulp also exhibited a higher C:N ratio than MSS (21:1 vs. 9:1), indicating greater carbohydrate availability and a more balanced nutrient profile. Lastly, the similar pH values in both substrates confirm the suitability of biopulp for anaerobic digestion applications and that would not inhibit the process.

**Figure 1a** shows the cumulative specific methane production and **Figure 1b** represents the TCOD degradation for the six trials over a 30-day period. All runs followed similar trends during the initial nine days, marked by rapid methane production, indicative of substrate availability. Furthermore, while the control (B-0) reached over half of the final TCOD degradation in 2 days, the co-digestion runs required 4 days to achieve similar levels, indicating a short adaptation phase. After 30 days, co-digestion trials achieved higher organic matter removal rates (ranging from 28% to 33% expressed as TCOD) than the control (25%). B-5 mixture achieved the highest methane yield (135)

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mL STP  $CH_4/g$   $TCOD_{added}$ ), a 14% higher than sewage sludge alone (118 mL STP  $CH_4/g$   $TCOD_{added}$ ). B-10 to B-30 had similar yields, still exceeding the control by 4%, indicating that greater biopulp volumes and organic loads did not hinder methane production. Statistical analysis confirmed significant performance differences, with the control consistently underperforming compared to biopulp-enhanced treatments. Co-digestion systems demonstrated increased stability and robustness, maintaining superior degradation performance throughout the digestion process.



 $\textbf{Figure 1} \ \textbf{Cumulative methane potential per g TCOD}_{\textbf{added}} \textbf{(a)}; \ \textbf{Normalized TCOD evolution (b)}.$ 

**Figure 2a** illustrates the evolution of SCOD and VFA, expressed as g COD/L, throughout the experiment while **Figure 2b** shows the ammoniacal nitrogen and pH evolution. Co-digestion samples (from B-5 to B-30) exhibited initial SCOD values of 5 (1) % relative to TCOD, compared to only 3% in B-0. Increasing biopulp volume led to higher SCOD concentrations as well as VFA. Results indicate that hydrolysis did not occur simultaneously across all treatments. Notably, B-5 and B-15 initiated acidogenesis one day earlier than the others, as evidenced by significant VFA accumulation on day 1 accompanied by a pH decrease. By the end of the experiment, B-0 showed a removal rate of 54% of SCOD while co-digestion treatments achieved 73 (3) % removal regardless of biopulp volume, indicating enhanced COD bioavailability that facilitated its degradation. These results confirm that biopulp, pretreated via thermal hydrolysis, provides a pre-hydrolyzed substrate enriched in SCOD, primarily in the form of VFA, readily available from the onset through day 9 of the experiment. This enhanced solubilization promotes acidogenesis, improving overall anaerobic digestion performance.

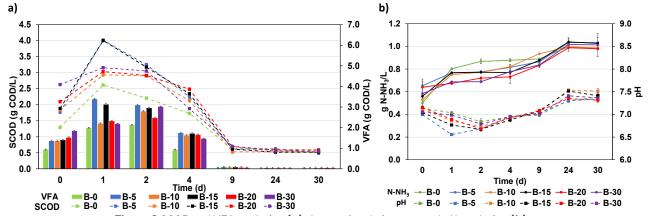


Figure 2 SCOD and VFA evolution (a); Ammoniacal nitrogen and pH evolution (b).

Maximum final ammoniacal nitrogen concentrations remained below inhibition thresholds (1.7 g N-NH $_3$ /L), without hindering microbial growth. Likewise, alkalinity, reached values  $\geq 2.5$  g CaCO $_3$ /L, improving system stability. In co-digestion, pH dropped to  $6.8 \pm 0.1$  over the first two days, boosting acidogenesis and briefly limiting methanogenesis due to early VFA and ammonia buildup.



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#### Conclusion

Thermal hydrolysis significantly boosted organic matter bioavailability by breaking down complex compounds and releasing soluble substrates that supported digestion. Co-digestion enhanced methane production by 4–14% per unit of TCOD added compared to mixed sludge, with no negative effects from increasing biopulp volume. All co-digestion treatments followed methanogenic pathways similar to sewage sludge digestion, although improved by greater initial availability of biodegradable organic matter. Treatment B–5 (95% mixed sewage sludge, 5% biopulp) achieved the highest methane yield, confirming co-digestion with thermal hydrolysis as an effective strategy to optimize anaerobic digestion performance.

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