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Ultrasound Treatment of Digested Sludge: a dual approach for Energy Recovery and Emerging Contaminant Reduction

CIOTOLA E.*[1], ESPOSITO G.[1], PIROZZI F.[1], CESARO A.[1]

[1]Department of Civil, Architectural and Environmental Engineering, University of Napoli Federico II, via Claudio 21, 80125, Napoli, Italy

*corresponding author: Enrica Ciotola

e-mail: enrica.ciotola@unina.it

Abstract Digested sludge (DS) is a valuable matrix that still holds potential for energy and nutrient recovery. This study explored the application of low-frequency ultrasound (US) treatment of DS to enhance organic matter solubilization and Polycyclic Aromatic Hydrocarbons (PAHs) transfer while reducing energy losses on already biodegradable substances. US treatment significantly increased solubilization, enhancing the specific methane production (SMP) by 51%. It also mobilized PAHs into the liquid phase. Preliminary BMP tests showed an improvement in the removal of target compounds, highlighting US as a promising method for improving DS biodegradability and quality.

Keywords: Anaerobic digestate; Ultrasonic treatment; Emerging contaminants; Recovery; Circular economy

1. Introduction

Anaerobic digestion (AD) is a well-established process for stabilizing organic matter in sludge (Zhang et al., 2024). A major advantage of AD lies in the recovery of energy as methane, alongside the production of a nutrient-rich byproduct, the DS (Di Costanzo et al., 2023). Enhancing AD efficiency, both in terms of biogas production and DS quality, supports the broader goals of resource recovery and waste minimisation (Ruiz-Hernando et al., 2022). In this context, low-frequency US treatment has emerged as a promising technique to improve sludge biodegradability, boost methane yields (Pilli et al., 2011) and facilitate the removal of trace organic contaminants by promoting their desorption from solid phases and enhancing their susceptibility to degradation (de Andrade et al., 2021). This study investigates, for the first time, the effects of US treatment on DS with a simultaneous focus on organic matter solubilization, biomethane potential enhancement, and the removal of target emerging contaminants, namely PAHs. Directing the energy input towards the digestate, which retains recalcitrant organic matter unconverted during AD, may further improve process efficiency by maximizing biogas recovery from the residual fraction.

2. Materials and Methods

2.1. Digestate sampling and characterization

DS was collected from the Salerno Sistemi SpA wastewater treatment plant (Salerno, Italy). Due to the low concentration of PAHs, it was spiked with a mix of the 16 PAHs listed as priority pollutants by the Environmental Protection Agency (EPA) at environmentally relevant concentrations. **Table 1** shows the main physico-chemical characteristics of the sampled DS, together with the initial and spiked concentrations of the sum of the 16 target PAHs under evaluation.

Table 1. ADS characterization

Parameter	Unit	Value
sCOD	[g_{COD}/L]	0.2 ± 0.0
tCOD	[g_{COD}/L]	28.6 ± 1.5
pH	[-]	7.3
VFAs	[$g_{CH3COOH}$/L]	0.02 ± 0.0
Alkalinity	[g_{CaCO3}/L]	2.1± 0.0
Σ 16 EPA PAHs before spiking	[µg/g_{DW}]	0.4
Σ 16 EPA PAHs after spiking	[µg/g_{DW}]	2.5

2.2 Sonication and BMP Tests

Sonication was performed using a SONOPULS Ultrasonic homogeniser (200 W, 20 kHz frequency) with a titanium probe (13 mm diameter) at 100% amplitude. The probe was immersed 2 cm into 350 mL volume of DS in a 60x170 mm cylindrical vessel. Three different treatment times were tested (2,5 and 10 minutes).

BMP tests were conducted in batch mode at 37°C. The inoculum was mixed with the treated DS at a (1:1) volume ratio. Two different controls were included to evaluate both the methane production from the inoculum and the untreated contaminated DS.

2.3 Analytical Methods

Total and soluble COD, total solids (TS), volatile solids (VS), and alkalinity were measured following Standard Methods. Soluble proteins (sC) and carbohydrates (sC) were quantified using the Lowry and Dubois methods, respectively. Volatile Fatty Acids (VFAs) were analyzed by High-Performance Liquid Chromatography. PAHs were extracted both from the solid and the liquid phase of

DS and analysed by Gas Chromatography-Mass Spectrometry (GC-MS).

3. Results and Discussion

Increasing US energy input resulted in enhanced disruption of the solid DS particles, releasing organic components into the liquid phase. This was confirmed by a 260% increase in sCOD content. Additionally, a significant increase in the VFAs concentration was observed, up to 3,698 $mg_{CH3COOH}/L$. This phenomenon can be attributed both to the breakdown of long-chain carboxylic acids triggered by the implosion of cavitation bubbles and the oxidative reactions involving the free radicals and the organic constituents of DS. Sonication did not significantly alter the total PAH concentration, but it notably shifted their distribution between the solid and liquid phases. Interestingly, the treatment promoted the mobilization of PAHs into the supernatant, with the increase in the sum of the 16 PAHs concentration in the 0.45 µm filtered liquid phase and a reduction of the overall concentration in the solid phase. Methane production was significantly enhanced in US-treated ADS, with similar specific methane production (SMP) for 2 and 5-min treatment and the highest SMP observed after 10-min treatment. This suggests that the improved bioavailability of organic matter directly enhanced methanogenesis. **Figure 1** shows the SMP increase observed under the different US conditions compared to untreated DS. Over 34 days of AD, sC initially declined rapidly. On the contrary, sP degradation was slower, indicating the release of more complex, less biodegradable proteins. Moreover, PAHs transfer to the supernatant potentially improved the biodegradation of some of the target PAHs during the re-digestion of the US-treated DS, but conflicting results were obtained depending on the target molecule. Notably, statistically significant removal improvements for acenaphtylene and benzo[g,h,i]perylene, respectively, were observed under the most intense US treatment condition, consistently with their highest detected concentrations in the liquid phase.

4. Conclusions

This study demonstrates that US treatment enhanced DS biodegradability, improving SMP from 37 to 51%. It also increased PAH mobility in the supernatant, boosting ACY and B[ghi]PY removal during re-digestion under intensive treatment. US treatment of DS offers promising potential for energy and nutrient recovery in a circular economy framework. These findings must be interpreted cautiously, as apparent removal and sequestration phenomena may complicate the assessment of PAHs fate. Future research should focus on reproducibility and scaling up, to optimize mass balance, energy recovery, and disposal cost reductions through improved DS quality.

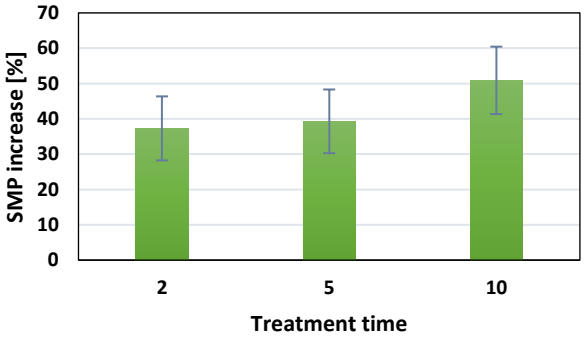

Figure 1. Effect of the US treatment on SMP production compared to untreated sludge.

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