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Biomethane Production Through Performance Enhancement of Anaerobic Digestion and Sludge Dewatering Processes in a Wastewater Treatment Plant

Tushar Kanti Sen [1*)], Anteneh Mesfin Yeneneh [2)]

[*1)]*Chemical Engineering Department, King Faisal University, P.O. Box 380, Al-Ahsa 31982 Saudi Arabia.*,
[2)]*International Maritime College, National University of Science and Technology, P.O. Box 532, Sohar, Oman*

Abstract

Sewage sludge management poses a persistent challenge due to increasing volumes and stringent environmental regulations. Current municipal wastewater treatment technologies are energy intensive, produce large quantities of residuals and fail to recover the potential resources available in wastewater. Anaerobic digestion (AD) is widely recognized for its cost-effective stabilization, high organic matter removal, and biogas production potential. However, the hydrolysis phase remains a bottleneck in AD, limiting methane yield. Furthermore, its application for low strength municipal wastewater is limited. To address such challenges, the effects of combined microwave-ultrasonic and combined microwave-oxidative pre-treatment of thickened excess activated sludge and mixed sludge on the performance enhancement of conventional mesophilic digester and on two-stage digesters are presented in this study. Microwave-ultrasonic pretreatment was optimized for enhanced cell disruption, while microwave oxidation pretreatment incorporated hydrogen peroxide to generate reactive oxygen species for accelerated organic solubilization. Experimental setups included single- and two-stage anaerobic digesters operated under mesophilic and thermophilic conditions. Results demonstrated that combined microwave-ultrasonic pretreatment improved cumulative methane production by 18% and total solids reduction by 31%, compared to microwave-only pretreatment. Microwave oxidation pretreatment with 1% H_2O_2 achieved 40% volatile solids solubilization, significantly enhancing methane yield and sludge dewaterability. The two-stage AD process further improved performance, achieving 76.4 mL/g TCOD methane yield and 58% methane content in biogas, outperforming single-stage systems. Findings indicate that hybrid pretreatments synergistically enhance hydrolysis, solubilization, and biogas production, making them promising solutions for sustainable sludge management in wastewater treatment plants.

Keywords: pretreatment, microwave-ultrasonic, microwave-oxidation, thermophilic, Bio methane.

Paper type: Research paper

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Introduction

Sewage sludge management and disposal remain significant challenges in the operation of municipal wastewater treatment plants. The production of waste-activated sludge has increased due to the growing number and capacity of these plants. Stricter environmental regulations and social constraints have made sludge disposal more difficult, and the capacity of these facilities. However, stricter environmental regulations and societal constraints have made sludge disposal more complex, while technologies such as incineration are often cost-prohibitive (Navaratnam, 2007). Anaerobic digestion for biogas production has gained worldwide attention due to its numerous benefits. These include eco-friendly energy generation, reduction of greenhouse gas emissions, efficient stabilization of large volumes of diluted organic matter at low cost, significant organic matter removal, minimal biomass production, rapid destruction of pathogenic microorganisms, and the production of a solid residue suitable for use as a soil conditioner.

*Corresponding author: E-mail: tsen@kfu.edu.sa; anteneh@imco.edu.om
ORCID: https://orcid.org/0000-0003-3085-5600

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Furthermore, anaerobic digestion can reduce feedstock odour by up to 80% (Ghosh *et al.*, 1975) and is widely regarded as one of the most energy-efficient and environmentally beneficial bioenergy technologies (Chong *et al.*, 2012; Müller, 2001; Weiland, 2010). Compared to aerobic digestion, anaerobic processes are preferred due to their lower costs, reduced energy consumption, and comparable performance (Appels *et al.*, 2008). Anaerobic digestion consists of four main microbial degradation steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. The hydrolysis phase is often the rate-limiting step due to the presence of extracellular polymeric substances (EPS) in excess sludge, which resist degradation and slow the overall process (Chong *et al.*, 2012; Tiehm *et al.*, 1997). A sustainable approach to sludge management involves minimizing sludge production while maximizing methane yield. To enhance hydrolysis, biodegradability, and dewaterability, various sludge pretreatment methods have been investigated, including ultrasonic (Apul and Sanin, 2010; Farooq *et al.*, 2009; Saifuddin and Fazlili, 2009; Yeneneh *et al.*, 2017), chemical (Gogate and Pandit, 2004; Eskicioglu *et al.*, 2007; Park, 2011; Yuan *et al.*, 2019), microwave (Yeom *et al.*, 2002; Liu *et al.*, 2017), ozone oxidation (Yuan *et al.*, 2023; Dai *et al.*, 2023), and other mechanical and thermal disintegration techniques (Tan *et al.*, 2020).These pretreatment methods primarily focus on disrupting cell walls and breaking down complex organic molecules. Ultrasonic pretreatment, for instance, utilizes cavitation-induced hydro-mechanical shear forces to degrade macromolecules with molecular weights exceeding 40,000 (Yeneneh *et al.*, 2017). The efficiency of ultrasonic treatment depends on factors like energy input, ultrasonic frequency, and sludge properties. Higher frequencies enhance radical oxidation, while lower frequencies generate mechanical and physical effects such as pressure waves (Lin and Lee, 2002). Ultrasonic pretreatment has been widely adopted in wastewater treatment plants (WWTPs) (Tan *et al.*, 2020). Similarly, microwave (MW) irradiation has proven effective in improving sludge biodegradability, methane production, and digestion efficiency (Liu *et al.*, 2017). Compared to conventional heating, MW pretreatment enhances biogas production, reduces sludge viscosity, improves dewaterability, and accelerates pathogen decay (Yeom *et al.*, 2002). For example, MW pretreatment has been shown to increase the soluble chemical oxygen demand (SCOD) to total chemical oxygen demand (TCOD) ratio from 2% to 22% (Eskicioglu *et al.*, 2008). Combining microwave and ultrasonic pretreatment offers a fast and cost-effective solution for improving sludge digestibility and biogas production. Studies have reported significant enhancements in gas production, solid removal, and dewaterability when these technologies are used together compared to their standalone application (Saifuddin & Fazlili, 2009; Yuan *et al.*, 2023). This combination provides synergistic effects, such as thermal and mechanical sludge disintegration, floc destruction, and cell wall rupture, resulting in greater organic release. Advanced oxidation processes (AOP) also play a critical role in sludge pretreatment. Their effectiveness depends on the generation rate of free radicals and their interaction time with organic molecules (Yeom *et al.*, 2002; Yuan *et al.*, 2023). For instance, microwave-H_2O_2 (MW-H_2O_2) and thermal-H_2O_2 hybrid treatments have been studied due to their ability to enhance biogas production and sludge solubilization (Yeom *et al.*, 2002; Yuan *et al.*, 2019). While early research on MW-H_2O_2 pretreatment yielded mixed results, recent studies suggest that optimizing H_2O_2 dosage is crucial for improving biogas production and minimizing refractory compound formation (Tan *et al.*, 2020). These findings underscore the potential of hybrid MW-H_2O_2 treatments to boost sludge solubilization and biomethane generation. However, the integration of hybrid MW-H_2O_2 pretreatment with two-stage anaerobic digestion remains largely unexplored. Moreover, the precise mechanisms by which reactive oxygen species (ROS) generated during MW-H_2O_2 pretreatment enhance sludge breakdown and subsequent anaerobic digestion require further investigation. This study aims to evaluate the combined effects of microwave-ultrasonic and microwave oxidation pretreatments on the anaerobic digestibility of thickened excess activated sludge and mixed sludge. It explores improvements in biogas production, solid removal, COD reduction, and sludge dewaterability, building on prior evidence of significant enhancements with combined pretreatment. The research seeks to ensure higher biogas yields, improved dewaterability, and overall enhanced performance in wastewater treatment plants (WWTPs)

1 Materials and methods

1.1 Sludge sampling and characterization

Primary sludge (PS) was obtained from the underflow of primary sedimentation tank No. 4 at the Beenyup Wastewater Treatment Plant (BWWTP). Thickened excess activated sludge (TEAS) was sourced from the discharge of the dissolved air flotation tank (DAFT) before mixing with the primary sludge. The final sludge mixture, comprising 75% PS and 25% TEAS, was used as feedstock for pretreatment and subsequently introduced into jacketed digesters. Characterization of the sludge samples included measurements of total solids (TS%), volatile solids (VS%), total chemical oxygen demand (TCOD), soluble chemical oxygen demand (SCOD), and pH after pretreatment.

1.2 sludge preparation and pretreatment

Microwave pretreatment was chosen for its advantages, including rapid and efficient heating, energy efficiency, non-contact application, and athermal effects, making it a highly effective method. The combination of microwave and ultrasonic treatments was also investigated due to their complementary effects, which enhance heat and mass transfer. Optimal conditions for combined microwave-ultrasonic pretreatment were determined based on prior research to ensure maximum performance. Sludge in digester 1 underwent microwave pretreatment, while sludge in digester 2 was subjected to combined microwave-ultrasonic pretreatment. For hybrid microwave-H_2O_2 (MW-H_2O_2) pretreatment, optimized microwave conditions (power and duration) and the ideal H_2O_2 dosage were applied to both single-stage and two-stage anaerobic digestion. To account for the impacts of catalase activity and residual H_2O_2, a sequential treatment approach (MW-H_2O_2-MW) was adopted (Wang *et al.*, 2009). The equipment and operational parameters for microwave and H_2O_2 hybrid heatments. The equipment and operational parameters for microwave and H_2O_2 hybrid treatments are similar to those outlined in section 1.3 and 1.4 respectively.

1.3 Experimental design and digester setup

The study assessed the anaerobic biodegradability of mixed sludge using microwave pretreatment and combined microwave-ultrasonic pretreatment, as depicted in **Figure 1** (a). In digester 1, 0.5 L of mixed sludge was treated using microwave irradiation at 2450 MHz with an energy density of 3.2W/mL for 10 minutes before being fed into a semi-batch continuously stirred anaerobic digester operating at an organic loading rate of 2.75g COD/L per day. In digester 2, sludge was subjected to microwave pretreatment followed by ultrasonic treatment at a density of 0.4W/mL, 90% amplitude, 150W intensity, and a pulsed mode of 55/5 for 8 minutes. Both digesters were purged with N_2 gas at a flow rate of 25–40mL/min, and mesophilic semi-batch anaerobic digestion was carried out for 45 days. A jacket heating system maintained a stable digester temperature of 36.5°C, while magnetic stirrers ensured continuous mixing. Biogas produced during the process passed through a 1 L buffering bottle to remove condensates, and measurements of gas volume and composition were taken over 20–25 days. A two-stage anaerobic digestion system, shown in Fig. 1(b), was set up using two pairs of 1-liter reactors in a semi-continuous configuration. Reactors (a) and (c) formed one two-stage system, while reactors (b) and (d) constituted a control system. Reactors (a) and (b) served as phase-I digesters, operating under thermophilic conditions at 55 ± 2°C, while reactors (c) and (d) were phase-II digesters, maintained at mesophilic conditions at 37±2°C.Temperature control was achieved through water circulation in a jacket heating system. Sampling ports were included for collecting sludge and gas samples, and hydraulic retention times (HRTs) were managed using Masterflex LS peristaltic pumps.

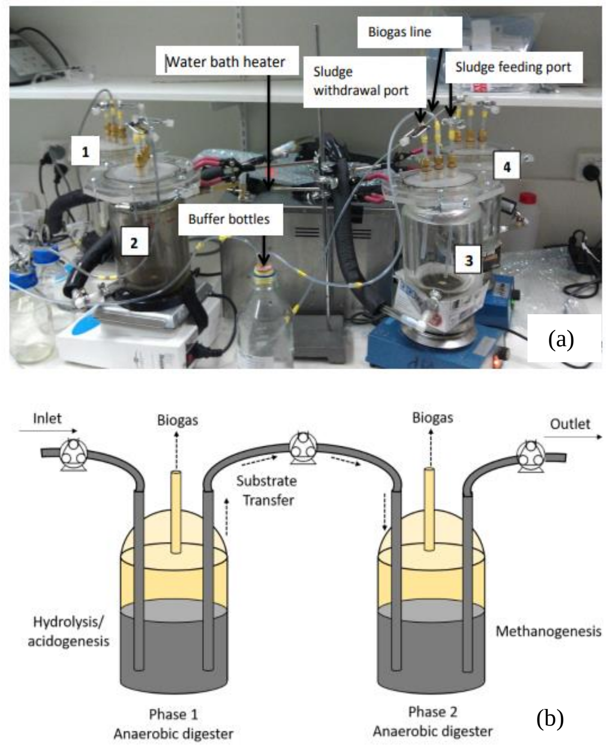

Fig. 1 (a) Combined Microwave-ultrasonic pretreatment-based anaerobic bioreactor setup,(b) microwave oxidation-based two-stage anaerobic bioreactor setup.

1.4 Analytical methods

A range of analytical methods was employed to evaluate sludge properties and digestion performance. These included analyses of VS, TS, TCOD, SCOD, pH, dewaterability, particle size, elemental composition. pH and temperature were measured using WP-90 and WP-81 TDS-pH/conductivity/temperature sensor. COD was determined using HACH reagents and colorimetric analysis with an ORION UV/vis spectrophotometer. SCOD was determined after centrifuging sludge samples at 5000 rpm for 10 minutes and filtering the supernatant through Whatman filter paper. Dewaterability tests were performed on different sludge samples to assess improvements in handling properties.

2. Results and Discussion

2.1 Anaerobic digester performance for microwave and combined microwave ultrasonic pretreated sludge

The impact of microwave and combined microwave-ultrasonic pretreatment on cumulative methane production (mL/g TCOD) is illustrated in **Figure 2**. After a sludge retention time of 22 days, cumulative methane production for mixed sludge treated with combined microwave-ultrasonic pretreatment increased by 18% compared to microwave-pretreated sludge. Both pretreatment methods significantly boosted methane generation relative to untreated sludge, attributed to the rapid heating, effective heat penetration, and athermal effects of microwave treatment, along with the floc disintegration benefits of ultrasonic treatment.

Figure 3 highlights a 31.4% enhancement in TCOD removal for sludge subjected to combined microwave-ultrasonic pretreatment compared to microwave-only pretreatment. Total solids reduction was 37.7% for microwave-pretreated sludge and 69.1% for sludge treated with the combined approach. Similarly, volatile solids reduction was 37.3% for microwave-pretreated sludge and 58.4% for combined microwave-ultrasonic treated sludge. This translates to a 31% increase in total solids removal and a 21% improvement in volatile solids removal for the combined pretreatment method.

The combined microwave-ultrasonic pretreatment demonstrated a significant enhancement in sludge biodegradability. Microwave pretreatment effectively disrupted cells through rapid internal heating, while ultrasonic treatment promoted cavitation, reduced floc size, and generated highly reactive radicals, facilitating the breakdown of organic matter. The observed improvements in volatile solids removal and SCOD solubilization were directly reflected in increased methane production and enhanced digestion kinetics.

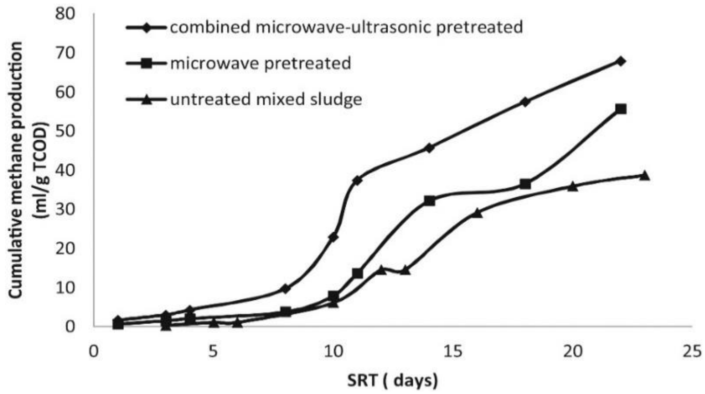

Fig. 2 Total methane generation for untreated, microwave and combined microwave-ultrasonic pretreated mixed digested sludge (*Reproduced from Yeneneh et al, 2015 with written permission*).

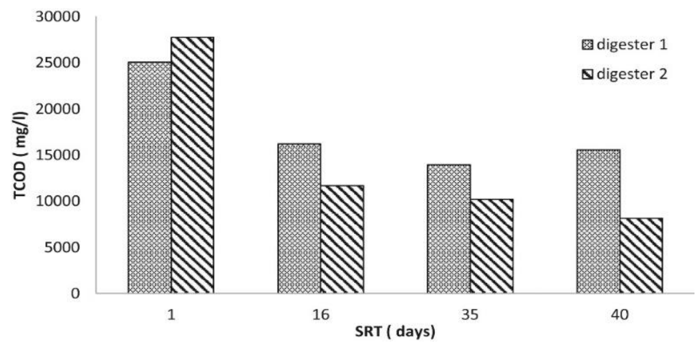

Fig. 3 (digester 1) TCOD reduction for microwave-treated and (digester 2) combined microwave-ultrasonic pretreated mixed sludge for varying SRT. (*Reproduced from Yeneneh et al, 2015 with written permission*).

2.2 Anaerobic biodegradability of microwave oxidation pretreated sludge

Microwave oxidation pretreatment significantly reduced total solids (TS) and volatile solids (VS), with total suspended solids (TSS) solubilization reaching saturation under combined treatment conditions, as depicted in **Figure 4** (a). However, further increases in microwave intensity and H_2O_2 concentration led to caramelization and the formation of refractory compounds. The effectiveness of advanced oxidation processes depends on the inactivation of the catalase enzyme before exposing the sludge to oxidative stress. Catalase facilitates the breakdown of hydrogen peroxide into water and oxygen, thereby protecting cells from radical formation under oxidative conditions. To address this, microwave treatment was applied before H_2O_2 treatment in this study to inactivate the catalase enzyme. The combined treatment achieved the highest VSS solubilization efficiency (40%) with 1% H_2O_2/TS. Treatments with 0.5% H_2O_2/TS and 1% H_2O_2/TS resulted in 19% and 33% VSS solubilization, respectively. The synergistic effects of the combined microwave-H_2O_2 pretreatment facilitated the generation of reactive species, such as hydroxyl and superoxide radicals, which significantly enhanced solubilization efficiency.

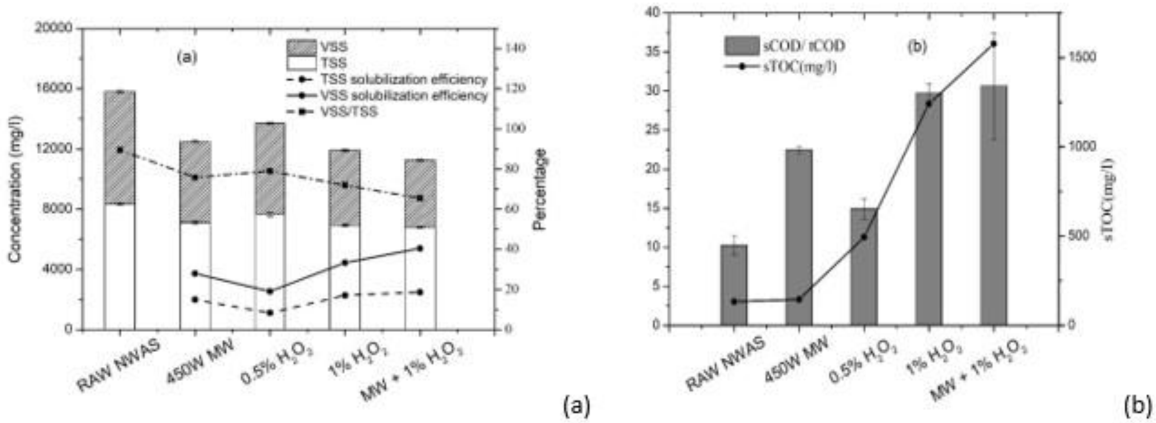

Fig. 4 (a) Effect of microwave oxidation pretreatment on NWAS solubilization in terms of total and volatile suspended solid removal, (b) Effect of microwave oxidation pretreatment on NWAS solubilization in terms of sTOC and sCOD removal.

2.3 Two stage anaerobic digestion process with microwave oxidation pretreatment

The performance of conventional single-stage (mesophilic) and two-stage (thermophilic-mesophilic) anaerobic digestion systems was evaluated by comparing cumulative methane production and biogas quality, as shown in **Figure 5**. The two-stage digester produced a cumulative methane yield of 76.4mL/g tCOD, significantly higher than the 40.4mL/g tCOD achieved by the single-stage mesophilic digester after 24 days of solid retention time (SRT) (Fig. 5(a)). In addition to higher methane yields, the two-stage digester exhibited superior biogas quality. Substrate transfer to the methanogenic reactor occurred on day 5, facilitating enhanced performance in phase II (Fig. 5(b)). The methanogenic reactor of the two-stage system maintained an average methane content of 51%, compared to the single-stage digester, which peaked at 49 ± 1.6% methane concentration between days 16 and 18. By day 10, the two-stage digester achieved a methane content of 58%, highlighting its efficiency in biogas quality improvement (Fig. 5(b)).

The accelerated hydrolysis of sludge in the two-stage digester enhances the activity of acidogenic bacteria, which break down complex organic matter into simpler volatile fatty acids (VFAs). These VFAs are then utilized by methanogenic bacteria for methane production during phase II. This process is evident from the VFA accumulation observed in both conventional single-stage and two-stage digesters, as shown in Fig 5. At the end of phase-I thermophilic digestion, VFA concentration was 2700 ± 60 mg/L, significantly higher than the levels in the single-stage mesophilic digestion at day 4 **Figure 6**. The organic acid conversion rate was lower in the single-stage digester, likely due to reduced solubilization. After 24 days of solid retention time (SRT), the single-stage digester achieved a 43% reduction in volatile solids (VS), while the two-stage digester achieved a higher VS reduction of 61%.

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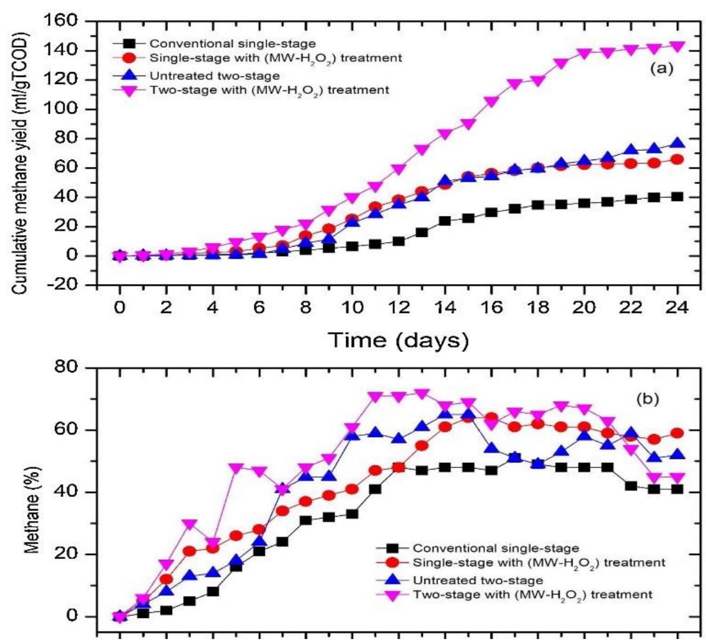

Fig. 5 anaerobic digestion performance of the four digester set-ups based on cumulative methane yield (ml/g) tCOD) (b) methane percentage. (*Reproduced from Ambrose et al., 2020 with written permission*).

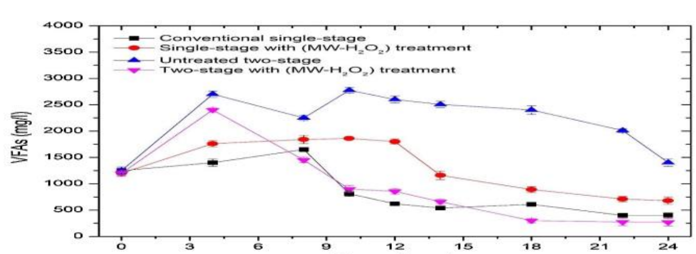

Fig. 6 Volatile fatty acids (VFAs) production from the four anaerobic digesters. Reproduced from Ambrose et al. 2020 with written permission

Conclusions

This study highlights the potential of advanced pretreatment techniques to overcome the limitations of anaerobic digestion, particularly the slow hydrolysis phase. The combined microwave-ultrasonic pretreatment demonstrated significant improvements in sludge disintegration, biodegradability, and methane production, with a 31% increase in total solids reduction and 18% higher methane yield compared to microwave-only treatment. Similarly, microwave oxidation pretreatment, particularly with 1% H_2O_2, showed enhanced volatile solids solubilization (40%) due to the generation of reactive oxygen species. The two-stage anaerobic digestion system further amplified these benefits, achieving superior methane yields and higher biogas quality compared to single-stage systems. The thermophilic-mesophilic configuration facilitated rapid hydrolysis and efficient conversion of volatile fatty acids into methane, demonstrating a robust strategy for improving sludge stabilization. These findings underscore the viability of integrating hybrid pretreatment methods with two-stage AD systems for optimizing sludge management. By enhancing biodegradability and methane production, these approaches align with sustainable energy goals while addressing the challenges of sludge disposal in municipal wastewater treatment plants. Further studies could focus on scaling up these technologies and exploring the mechanisms of reactive species generation and their interaction with sludge organics.

Nomenclature

AD	=Anaerobic digestion	[-]
AOP	=Advanced oxidation processes	[-]
EPS	=Extracellular polymeric substances	[-]
MW	=Microwave	[-]
PS	=Primary sludge	[-]
TEAS	=Thickened excess activated sludge	[-]
DAFT	=Dissolved air flotation tank	[-]
TS %	=Percentage total solids	[mg/l]
TSS	=Total suspended solids	[mg/l]
VS %	=Volatile solids	[mg/l]
VSS %	=Percentage volatile suspended solids	[mg/l]
TDS	=Total dissolved solids	[mg/l]
TCOD	=Total chemical oxygen demand	[mg/l]
SCOD	=Soluble chemical oxygen demand	[mg/l]
VFA	=Volatile fatty acids	[mg/l]
SRT	=Solid retention time	[day]
HRT	=Hydraulic retention time	[day]

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