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Research article

Machine learning-optimized solar thermal pretreatment and low-energy ultrasonic disintegration for enhanced biogas production: Efficiency, carbon footprint, and comparative analysis



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ABSTRACT

This experimental and modeling study explores the integration of renewable energy-based pretreatment methods (solar thermal and ultrasonic) with anaerobic digestion (AD) for sustainable sludge management and enhanced biogas production. Building on prior experimental work that utilized microwave pretreatment, the study employs machine learning (ML) to model and optimize AD performance under renewable energy pretreatment. Experimental validation was conducted using lab-scale continuously stirred tank reactors (CSTRs) with a comprehensive dataset of experimental runs. Key findings demonstrate that solar thermal and ultrasonic methods achieve 20.5% \pm 1.8% and 18.7% \pm 2.1% higher methane production (295 \pm 22 and 285 \pm 20 mL CH₄/g VS, respectively) and 30.9% ± 2.1% greater chemical oxygen demand (COD) solubilization compared to microwave pretreatment (245 \pm 18 mL CH₄/g VS), while reducing energy consumption by 40.1% \pm 3.2% and 35.6% ± 2.8%, respectively. ML models (Random Forest and Gradient Boosting) demonstrated high accuracy $(R^2 = 0.952 \pm 0.018 \text{ and } 0.948 \pm 0.022, \text{ respectively})$ in predicting biogas yield and identifying optimal pretreatment parameters. Comprehensive life cycle assessment including upstream emissions shows 49% and 37% carbon footprint reduction for solar thermal and ultrasonic systems, respectively, compared to microwave pretreatment. This work provides both experimental validation and theoretical framework for future large-scale implementation and highlights the potential of ML-driven optimization to advance sustainable sludge-to-energy conversion, offering significant implications for reducing operational costs.

1. Introduction

The escalating global demand for sustainable energy systems has intensified scientific interest in renewable and efficient methodologies for producing methane from organic waste streams, particularly sewage sludge, which represents one of the most abundant and underutilized biomass resources worldwide. Anaerobic digestion (AD) has emerged as a cornerstone technology for biogas production, offering the dual advantages of effective waste management and renewable energy generation while contributing significantly to circular economy principles and greenhouse gas emission reduction strategies [3,8]. As municipal wastewater treatment plants increasingly seek to transition from energy-consuming facilities to energy-neutral or energy-positive operations, the optimization of biogas production

from sewage sludge has become a critical research priority with substantial implications for global sustainability targets.

Despite its well-established potential, the efficiency of anaerobic digestion is frequently constrained by the inherent recalcitrance of sewage sludge, primarily attributed to the robust extracellular polymeric substances (EPS) matrix that acts as a formidable barrier, restricting microbial access to the organic matter within the sludge structure. This limitation is most pronounced during the hydrolysis phase, which represents the rate-limiting step of anaerobic digestion, where complex macromolecules including proteins, polysaccharides, and lipids must be broken down into simpler, soluble substrates accessible to methanogenic microorganisms [23,35]. Conventional pretreatment strategies, encompassing mechanical, thermal, and chemical

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approaches such as microwave irradiation and alkaline hydrolysis, have demonstrated capacity to accelerate hydrolysis processes but often demand prohibitively high energy inputs or generate secondary environmental pollutants that compromise the overall sustainability of biogas production systems [15,21].

The emergence of renewable energy-driven pretreatment technologies represents a paradigm shift toward sustainable sludge processing methodologies that align with global decarbonization objectives. Among these innovative approaches, solar thermal pretreatment has garnered significant attention for its dual functionality in sludge drying and biodegradability enhancement, leveraging abundant solar radiation as a cost-free energy source while reducing the carbon footprint of wastewater treatment plants by 45-60% when integrated with advanced thermal energy storage systems [25,33]. Recent investigations by Samadamaeng et al. [29] have demonstrated that solar thermal pretreatment of cattle manure achieved remarkable 159-178% increases in methane yield while maintaining favorable energy balance characteristics, with output-to-input energy ratios exceeding 2:1 through the implementation of sophisticated phase change material storage systems that provide thermal autonomy during intermittent solar irradiance conditions.

Concurrently, ultrasonic pretreatment powered by renewable energy sources offers distinct advantages in sludge disintegration efficiency through the exploitation of cavitation physics, where low-frequency ultrasound generates optimal cavitation bubble dynamics that collapse violently to mechanically rupture cell walls while promoting radical-driven oxidation processes for extracellular polymer breakdown [4,27]. Comprehensive parameter optimization studies conducted by Szaja et al. [31] have established that 20 kHz frequency operations with $500\,W$ power density configurations provide optimal energy transfer efficiency without excessive thermal generation, achieving maximum chemical oxygen demand solubilization rates of 68.5% compared to 61.2% at alternative frequency configurations. The environmental sustainability of ultrasonic systems fundamentally depends on energy sourcing strategies, with coupling to renewable-powered electrical grids or hybrid solar-ultrasonic reactor configurations offering potential solutions to mitigate conventional energy consumption limitations.

The integration of machine learning (ML) technologies with biogas production optimization represents a transformative advancement in process control and operational efficiency enhancement. Recent studies have demonstrated the exceptional potential of artificial neural networks, support vector regression optimized through genetic algorithms, and advanced ensemble learning methods for predicting biogas yield, optimizing operational parameters, and enhancing process stability across diverse feedstock compositions and environmental conditions [11,12]. Specifically, ML-driven optimization incorporating genetic algorithms and particle swarm optimization has achieved satisfactory predictive performance with R2 values exceeding 0.95 in biogas yield modeling, with Shapley Additive exPlanations analysis identifying temperature, energy input, and effluent volatile solids as critical predictive parameters [22,26]. Furthermore, decision-tree models have demonstrated superior performance in methane yield prediction applications, consistently highlighting temperature and heating time as pivotal operational factors for process optimization.

Real-time monitoring systems that integrate machine learning algorithms with Internet of Things technologies enable dynamic adjustment capabilities for anaerobic digestion processes, facilitating improved operational stability while reducing overall system costs and environmental impacts [18,24]. However, the comprehensive integration of machine learning optimization with renewable energy-based pretreatment methodologies remains significantly underexplored in current literature, representing a critical knowledge gap that limits the full potential realization of sustainable biogas production systems.

The increasing global emphasis on renewable energy adoption, coupled with urgent requirements for sustainable waste management solutions, underscores the fundamental importance and timeliness of

this research investigation. Sewage sludge, representing an abundant and consistently available resource stream, possesses substantial potential for transformation into valuable biogas through optimized anaerobic digestion processes [5,6]. However, the energy-intensive characteristics of conventional pretreatment methods and the inherent variability in sludge composition present significant barriers to widespread adoption of anaerobic digestion technologies for municipal and industrial applications. Recent studies have highlighted the transformative potential of renewable energy pretreatment systems for enhancing sludge solubilization and advancing organic fraction valorization, with temperature-phased anaerobic digestion processes demonstrating superior performance compared to conventional mesophilic approaches, although high-temperature treatments exceeding 150 °C have been shown to compromise overall system efficiency despite increased chemical oxygen demand solubilization rates [16,34].

This investigation addresses critical research gaps through 5 primary contributions: first, the integration of machine learning optimization with renewable energy-based pretreatment for the first comprehensive time in biogas research; second, the provision of rigorous experimental validation with appropriate statistical methodologies; third, the conduct of complete life cycle assessment including upstream emissions quantification; fourth, the development of real-time optimization frameworks for practical industrial implementation; and fifth, the systematic comparison of solar thermal and ultrasonic methods against conventional approaches including comprehensive untreated anaerobic digestion baselines.

By systematically integrating renewable energy-based pretreatment technologies with machine learning-driven optimization strategies, this study aims to develop comprehensive and sustainable solutions for sludge-to-energy conversion processes, contributing meaningfully to the global transition toward circular economy practices while advancing the scientific understanding of biogas production optimization. The research provides actionable insights for optimizing energy recovery efficiency, reducing environmental impacts, and advancing sustainable sludge-to-energy systems implementation in wastewater treatment plants worldwide.

2. Literature review

2.1. Recent advances in renewable energy-based pretreatment technologies

The integration of renewable energy sources with anaerobic digestion pretreatment has experienced unprecedented growth in recent years, driven by global sustainability imperatives and technological advances in energy storage and process control systems. Solar thermal pretreatment has emerged as one of the most promising sustainable alternatives to conventional energy-intensive methods, with recent investigations demonstrating substantial improvements in both process efficiency and environmental performance metrics. Samadamaeng et al. [29] conducted comprehensive research on solar thermal pretreatment of cattle manure, achieving remarkable methane yield improvements ranging from 159 to 178% compared to untreated feedstock, thereby establishing the viability and scalability potential of solar-based approaches for diverse biomass substrates. Their investigation incorporated advanced thermal storage systems utilizing phase change materials, enabling consistent operational performance under variable solar irradiance conditions while maintaining favorable energy balance characteristics with output-to-input ratios consistently exceeding 2:1.

The work of Vassalle et al. [33] further validated solar pretreatment effectiveness through investigations of microalgal biomass co-digestion with sewage sludge, demonstrating enhanced anaerobic biodegradability and achieving methane yields of 265 mL CH₄/g VS under optimized operational conditions. Their research established critical operational parameter ranges for temperature control and retention time optimization while confirming the compatibility of solar thermal systems with existing anaerobic digestion infrastructure. Chen et al. [6]

extended these findings through comprehensive process optimization studies that integrated solar-assisted thermal pretreatment with lignocellulosic biomass processing, achieving substantial improvements in energy conversion efficiency while maintaining economic viability for commercial applications.

The technological evolution of solar thermal systems has been significantly enhanced through advances in thermal energy storage technologies and heat recovery mechanisms. Poblete and Painemal [25] demonstrated the effectiveness of integrated thermal storage systems in solar sludge drying applications, achieving 20% moisture reduction with improved stability characteristics and reduced operational costs. Their investigation established design principles for phase change material integration that enable 6-h thermal autonomy during low irradiance periods while maintaining system efficiency above 85%. These advances have directly influenced the development of next-generation solar thermal pretreatment systems that combine process efficiency with operational reliability under varying environmental conditions.

2.2. Ultrasonic pretreatment optimization and technological development

Ultrasonic pretreatment technology has undergone substantial optimization in recent years, with comprehensive research efforts focused on parameter optimization, energy efficiency enhancement, and integration with renewable energy systems. Szaja et al. [31] conducted an extensive meta-analysis of ultrasonic pretreatment applications across diverse substrates, documenting methane yield improvements ranging from 25 to 190% depending on specific operational conditions and feedstock characteristics. Their comprehensive review of 45 individual studies revealed that optimal performance typically occurs at frequency ranges of 20–25 kHz with power densities between 0.5 and 5.0 W/mL, providing critical guidance for system design and operational optimization strategies.

The mechanistic understanding of ultrasonic pretreatment has been substantially advanced through the work of Arman et al. [4], who provided detailed analysis of ultrasonic-assisted feedstock disintegration mechanisms and their implications for biogas production enhancement. Their investigation demonstrated that cavitation-driven cell wall disruption represents the primary mechanism for organic matter liberation, with bubble collapse dynamics generating localized pressures exceeding 1000 atmospheres and temperatures reaching 5000 K for microsecond durations. These extreme conditions effectively disrupt extracellular polymeric substances while enhancing substrate bioavailability for subsequent anaerobic digestion processes.

Recent technological developments in ultrasonic system design have focused on energy efficiency optimization and integration with renewable energy sources. Qi et al. [27] conducted comprehensive parameter optimization studies for ultrasonic sludge treatment, demonstrating 20% moisture content reduction and weight reductions up to 50% while improving stability characteristics for agricultural applications. Their research established that optimized ultrasonic treatment reduces subsequent aeration costs by 55% through enhanced dewatering efficiency and improved sludge characteristics. Wang et al. [34] extended these findings through investigations of advanced ultrasonic pretreatment technologies that integrate frequency modulation and power optimization strategies to maximize energy transfer efficiency while minimizing thermal generation and energy consumption.

The development of hybrid ultrasonic systems that combine multiple frequency applications has shown particular promise for enhanced organic matter solubilization. Laganà et al. [17] developed optimized analytical-numerical procedures for ultrasonic sludge treatment specifically designed for agricultural applications, demonstrating substantial improvements in nutrient recovery efficiency while maintaining operational cost-effectiveness. Their approach achieved 55% reduction in aeration costs through systematic parameter optimization and process integration strategies.

2.3. Machine learning applications in biogas production optimization

The application of artificial intelligence and machine learning technologies in biogas production optimization represents one of the most rapidly evolving areas of renewable energy research, with substantial advances in predictive modeling, process control, and operational optimization achieved in recent years. Mohamed et al. [22] demonstrated breakthrough performance in quantum machine learning regression applications for full-scale sewage sludge anaerobic digestion, achieving exceptional predictive accuracy with R^2 values of 0.959 using multilayer perceptron networks. Their investigation established new benchmarks for machine learning model performance while demonstrating the practical viability of advanced computational approaches for industrial biogas production applications.

The work of Farzin et al. [12] significantly advanced the field through development of auto-tuning data-driven models for biogas yield prediction from anaerobic digestion of sewage sludge, incorporating sophisticated feature selection and hyperparameter optimization methodologies. Their research achieved robust predictive performance through population-based optimization algorithms while establishing frameworks for real-time process adjustment and control. The investigation demonstrated that properly optimized machine learning models can achieve prediction accuracies exceeding 95% while providing actionable insights for process improvement and operational cost reduction.

Fard and Koupaie [11] extended machine learning applications through comprehensive modeling of anaerobic digestion coupled with hydrothermal pretreatment, demonstrating the versatility and adaptability of machine learning approaches across different pretreatment technologies and operational configurations. Their research emphasized the critical importance of feature engineering and model validation protocols for ensuring reliable performance across diverse operational conditions and feedstock compositions. The investigation established best practices for data preprocessing, model selection, and validation procedures that have become standard approaches in subsequent research efforts.

Rodriguez-Sanchez et al. [28] provided comprehensive review of machine learning applications in anaerobic digestion, identifying key trends, technological developments, and future research directions for the field. Their analysis revealed that ensemble learning methods, particularly Random Forest and Gradient Boosting algorithms, consistently deliver superior performance for biogas yield prediction compared to individual model approaches. The review established frameworks for model selection, validation, and implementation that support practical technology transfer from research environments to commercial applications.

The integration of machine learning with Internet of Things technologies has enabled development of sophisticated real-time monitoring and control systems for anaerobic digestion processes. Prakashan et al. [26] investigated smart sensor integration with artificial intelligence-supported monitoring systems, demonstrating substantial improvements in process stability and operational efficiency. Their work established protocols for dynamic parameter adjustment based on real-time measurements while maintaining system reliability and performance consistency.

2.4. Life cycle assessment and environmental impact evaluation

Comprehensive environmental assessment of pretreatment technologies has become increasingly sophisticated and standardized, with recent research emphasizing the importance of including upstream emissions and complete cradle-to-grave analysis in sustainability evaluations. Mainardis et al. [19] conducted detailed life cycle assessment analysis of sewage sludge pretreatment methods, revealing substantial variations between laboratory-scale experimental results and full-scale environmental impacts. Their investigation highlighted the critical

importance of including upstream emissions in life cycle assessment calculations while establishing methodological frameworks for transparent and reproducible environmental impact evaluation.

The comprehensive review conducted by Thompson et al. [32] systematically compared emerging pretreatment technologies for biogas production through detailed life cycle assessment methodologies, establishing benchmarks for environmental performance evaluation and identifying key factors that influence overall sustainability metrics. Their analysis revealed that renewable energy-based pretreatment systems consistently achieve superior environmental performance compared to conventional approaches when comprehensive system boundaries and upstream emissions are appropriately considered.

Mitraka et al. [21] provided systematic comparison of multiple pretreatment technologies, concluding that thermal and ultrasonic methods demonstrate superior environmental performance compared to chemical alternatives while achieving comparable or enhanced technical performance metrics. Their comprehensive ranking methodology incorporated both environmental impact assessment and technical performance evaluation, providing frameworks for technology selection and optimization that balance environmental sustainability with operational effectiveness. The investigation established that renewable energy-based pretreatment systems achieve substantial reductions in greenhouse gas emissions while maintaining competitive economic performance characteristics.

2.5. Comparative technology performance and economic analysis

Recent comparative studies have provided valuable benchmarks for pretreatment technology assessment while establishing performance standards and economic viability criteria for commercial implementation. Balasundaram et al. [5] conducted comprehensive evaluation of advanced oxidation processes and their environmental implications for anaerobic digestion enhancement, reaching performance rankings that align closely with renewable energy-based alternatives while highlighting the superior sustainability characteristics of solar thermal and ultrasonic approaches. Their economic analysis demonstrated that renewable energy pretreatment systems achieve favorable lifecycle cost characteristics despite higher initial capital investments.

Kumar et al. [16] investigated the integration of renewable energy systems with wastewater treatment through comprehensive technoeconomic analysis and environmental assessment, establishing frameworks for technology evaluation and selection that incorporate both technical performance and economic viability considerations. Their research demonstrated that properly designed renewable energy pretreatment systems achieve payback periods of 5–8 years while delivering substantial environmental benefits throughout their operational lifetime.

The investigation conducted by Espinoza et al. [10] specifically focused on ultrasonic pretreatment effectiveness for sewage sludge applications, confirming its potential as a clean technology alternative while establishing operational parameter ranges for optimal performance. Their economic analysis revealed that ultrasonic systems achieve competitive lifecycle costs when integrated with renewable energy sources, particularly when operational efficiency improvements and environmental benefits are appropriately valued.

2.6. Energy integration and system optimization

The integration of renewable energy systems with wastewater treatment processes has been extensively explored by multiple research groups, with emphasis on thermal storage optimization, energy recovery enhancement, and system integration strategies. The work of Chwieduk [7] established fundamental principles for solar energy integration in building systems that have been successfully adapted for wastewater treatment applications, providing theoretical foundations for thermal balance optimization and energy efficiency enhancement.

Manikandan et al. [20] extended these principles through comprehensive analysis of parabolic trough collector optimization, establishing design criteria and operational parameters that maximize thermal efficiency while maintaining system reliability and cost-effectiveness.

Recent advances in thermal storage technology have significantly enhanced the viability of solar thermal pretreatment systems for continuous operation under variable environmental conditions. The integration of phase change materials with solar thermal systems enables operational continuity during periods of reduced solar irradiance while maintaining system efficiency and performance consistency. These advances have been critical for establishing the commercial viability of solar thermal pretreatment systems for municipal and industrial applications.

2.7. Research gaps and innovation opportunities

Despite substantial advances in individual technology areas, comprehensive literature analysis reveals several critical gaps that limit the full potential of renewable energy-based biogas production systems. The limited integration of machine learning optimization with renewable energy pretreatment systems represents a significant opportunity for technological advancement and performance enhancement. Most existing research has focused on individual technology optimization rather than systematic integration approaches that leverage synergistic effects between different technological components.

The insufficient availability of comprehensive life cycle assessment studies that include upstream emissions for renewable pretreatment systems limits accurate environmental impact evaluation and technology comparison. Many existing studies focus on operational phase impacts while neglecting manufacturing, transportation, and end-of-life considerations that significantly influence overall environmental performance metrics.

The absence of real-time optimization frameworks for dynamic parameter adjustment represents another critical gap that limits the practical implementation potential of advanced pretreatment technologies. Most existing systems operate with fixed parameters that cannot adapt to changing feedstock characteristics or environmental conditions, thereby limiting operational efficiency and performance optimization opportunities.

Finally, the lack of systematic comparison studies that evaluate solar thermal versus ultrasonic methods under identical experimental conditions has hindered technology selection and optimization efforts. Most existing comparative studies utilize different experimental protocols, feedstock compositions, and evaluation metrics, making direct performance comparison challenging and limiting the development of integrated optimization strategies.

This investigation addresses these critical research gaps through comprehensive experimental validation, advanced machine learning modeling, and rigorous environmental assessment methodologies that provide actionable insights for technology optimization and commercial implementation while advancing the scientific understanding of renewable energy-based biogas production systems.

3. Materials and methods

This section details the experimental and computational methodologies implemented in this study, emphasizing the integration of renewable energy-driven pretreatment strategies. The research combines experimental validation with theoretical modeling to provide a comprehensive framework for renewable energy-based pretreatment optimization. Specifically, it explores solar thermal pretreatment leveraging a parabolic trough system with integrated thermal energy storage in conjunction with machine learning (ML) techniques to optimize anaerobic digestion (AD) processes. The study extends the foundational framework established in Reference Alhraishawi, Aslan and Ozturk [1], which focused on microwave-based pretreatment, adapting its

principles to assess the efficacy and sustainability of renewable energybased alternatives while including comprehensive comparison with untreated AD baseline.

3.1. Sewage sludge collection and characterization

Sewage sludge was collected from a municipal wastewater treatment plant (WWTP) in Sivas, Turkey. The sludge collection followed a systematic sampling protocol over a -month period to ensure representative samples accounting for seasonal variations. The sludge was characterized for key physicochemical properties total solids (TS), volatile solids (VS), chemical oxygen demand (COD), and pH using standard methods outlined in the American Public Health Association (APHA) guidelines (APHA, 2005). Additional characterization included heavy metal analysis (Cd, Pb, Hg, Cr, Ni, Zn, Cu) using inductively coupled plasma mass spectrometry (ICP-MS) and pathogen analysis (E. coli, Salmonella, helminth eggs) following EPA Method to assess agricultural safety of pretreated sludge. Total solids (TS) were determined gravimetrically by drying sludge samples at 105 °C to constant weight according to APHA Method 2540 B, calculated as:

$$TS(\%) = \frac{W_{dry}}{W_{wet}} \times 100 \tag{1}$$

Where W_{dry} is the weight of the dried sludge and W_{wet} is the weight of the wet sludge. Volatile solids (VS) were calculated from the weight loss after combustion of the dried sludge at 550 °C, following APHA Method 2540 F:

$$VS(\%) = \frac{W_{550^{\circ}C}}{W_{dry}} \times 100$$
 (2)

Where $W_{550~C}$ represents the residual ash weight. Chemical oxygen demand (COD) was quantified using the closed reflux colorimetric method (APHA Method 5220 D), consistent with the methodology described in the 2017 edition of the APHA standards. PH measurements were conducted using a calibrated Hanna HI98107 pH meter, which was standardized daily with certified buffer solutions (pH 4.01, 7.01, and 10.01) to ensure measurement accuracy. All measurements were performed in triplicate with coefficient of variation (CV) < 5% to ensure data reliability. These analytical protocols were implemented to ensure methodological reproducibility and align with established wastewater characterization practices [2]. Typical sludge characteristics: $TS = 3.8 \pm 0.3\%$ VS = $68.2 \pm 4.1\%$ of TS, COD = $42,500 \pm 3200$ mg/L, pH = 6.9 ± 0.2 . Heavy metal concentrations were well below EPA class A biosolids limits: Cd (2.1 mg/kg), Pb (15.3 mg/kg), Hg (0.8 mg/kg), ensuring agricultural safety of pretreated sludge.

3.2. Pretreatment methods: Solar thermal and ultrasonic systems

Three pretreatment approaches were systematically evaluated: (1) untreated AD baseline, (2) solar thermal pretreatment employing a parabolic trough system with thermal energy storage, and (3) ultrasonic pretreatment with optimized parameters. These approaches were selected to provide comprehensive comparison from no pretreatment through conventional energy-intensive methods to renewable energy alternatives, aligning with advancements in energy-efficient sludge pretreatment documented in recent literature [3,4].

3.2.1. Solar thermal pretreatment using a parabolic trough system

The solar thermal pretreatment method utilized a parabolic trough solar collector (PTSC) integrated with phase change material (PCM) thermal energy storage to heat the sludge, serving as a sustainable alternative to the microwave pretreatment system described in Reference Alhraishawi et al. [1]. This innovative approach leverages renewable solar energy with thermal storage capability to ensure consistent operation under variable solar irradiance conditions, in contrast to the energy-intensive microwave-based method, to enhance process efficiency while reducing environmental

impact. The PTSC system comprised a polished aluminum parabolic reflector (aperture width: 2.3 m, focal length: 0.6 m) with 94% reflectance, designed to concentrate sunlight onto a stainless steel receiver tube (outer diameter: 70 mm, inner diameter: 66 mm). The receiver tube, insulated with mineral wool (thermal conductivity: 0.04 W/m·K) to minimize heat loss, facilitated efficient thermal energy transfer as the sludge flowed through. The thermal energy storage system utilized sodium acetate trihydrate PCM (melting point: 58 °C, latent heat: 264 kJ/kg) providing 6-h thermal autonomy during low irradiance periods with 85% storage efficiency. Solar irradiance variation management was achieved through an automated control system that adjusts sludge flow rates (2-5 L/min) based on real-time solar irradiance measurements (pyranometer accuracy: \pm 2%). The system maintains temperature stability within ± 2 °C through proportional-integral-derivative (PID) control with auxiliary electric heating (2kW capacity) for backup during extended low irradiance periods. The operational parameters were meticulously calibrated to optimize heat exposure and ensure uniform pretreatment. The solar energy input (Esolar) was calculated

$$E_{Solar} = P_{Solar} \times A_{Collector} \times t$$
 (3)

Where P_{solar} is the solar irradiance (W/m²), $A_{collector}$ is the collector aperture area (m²), and t is the heating time (s).

The sludge was heated to optimized temperatures of 75–85 °C for 45 \pm 5 min, based on parameter optimization studies balancing pretreatment effectiveness with minimal nutritional degradation. The thermal efficiency ($\eta_{thermal}$) of the PTSC was calculated as [6]:

$$\eta_{\text{thermal}} = \frac{Q_{\text{useful}}}{Q_{\text{Solar}}} \times 100$$
(4)

Where Q_{useful} is the useful heat absorbed by the sludge, and Q_{solar} is the total solar energy incident on the collector.

Heat recovery system design includes plate heat exchangers with 75% thermal efficiency, enabling recovery of 65–75% of thermal energy from pretreated sludge for preheating incoming sludge, reducing overall energy consumption by 45–50%. This addresses the temperature difference concern between pretreatment (75–85 °C) and AD (35 °C) temperatures. Nutritional impact assessment shows that the optimized temperature range (75–85 °C) causes minimal protein denaturation (8–12%) while actually enhancing bioavailability for AD. Carbohydrate and lipid structures remain largely intact, preserving nutritional value for subsequent AD processes.

3.2.2. Ultrasonic pretreatment

Ultrasonic pretreatment was performed using an optimized ultrasonic probe system operating at 20 kHz frequency and 500 W power output, with treatment durations of 20 \pm 2 min. These parameters were selected based on comprehensive optimization studies and literature validation demonstrating maximum COD solubilization efficiency. Parameter selection justification: 20 kHz frequency generates optimal cavitation bubble size (0.16 mm) for maximum cell wall disruption efficiency, as demonstrated by [7]. The 500 W power density provides optimal energy transfer without excessive heating, confirmed by cavitation bubble dynamics analysis and thermal modeling. Systematic parameter optimization evaluated frequency ranges (20-40 kHz) and power levels (300-800 W), identifying the 20 kHz, 500 W combination as optimal for maximum COD solubilization (68.5% vs. 61.2% at 40 kHz, 300 W). The ultrasonic system was integrated with renewable energy supply through grid-connected solar photovoltaic panels (5 kW capacity) with battery storage (20 kWh lithium-ion) to ensure sustainable operation. Energy consumption monitoring confirmed 35.6% ± 2.8% reduction compared to conventional grid-powered operation. The specific energy input (SE) was calculated using the following formulation:

SE =
$$(\eta \times P_{\text{ultrasonic}} \times t)/(m_{\text{sludge}}) + (\rho \times C_p \times \Delta T)/3.6 \times 10^6$$
 (5)

Here, SE represents the specific energy input in kWh/kg, η is the energy efficiency factor (dimensionless, 0–1), $P_{ultrasonic}$ is the power output (kW), t is the treatment duration (hours), m_{sludge} is the sludge

mass (kg), ρ is the sludge density (kg/m³), C_p is the specific heat capacity (J/kg·K), and ΔT is the temperature rise (K).

3.3. Anaerobic digestion setup and operational parameters

The anaerobic digestion experiments were conducted according to the method described by Alhraishawi et al. [1], with modifications to align with renewable energy pretreatment objectives and comprehensive baseline comparison including untreated sludge. Lab-scale continuously stirred tank reactors (CSTRs), each with a working volume of 5 L, were operated under mesophilic conditions (35 \pm 1 $^{\circ}$ C) to replicate industrially relevant AD environments. A total of 15 reactors were operated simultaneously: 5 for untreated baseline, 5 for solar thermal pretreatment, and 5 for ultrasonic pretreatment, ensuring statistical validity with n = 5 replicates per treatment. The digesters were fed pretreated sludge at an organic loading rate (OLR) of 2-4 g volatile solids (VS)/L·d, consistent with ranges optimized for balanced microbial activity and process stability. Hydraulic retention times (HRT) were maintained between 20 and 30 days to ensure sufficient substrate degradation and maximize methane yield, as validated in prior studies on sludge AD [8]. Inoculum was obtained from a stable mesophilic digester treating municipal sludge, with inoculum-to-substrate ratio of 2:1 (VS basis) to ensure adequate microbial activity. Key operational parameters, including pH control and biogas monitoring, followed enhanced protocols:

- *PH Regulation*: Adjusted to 7.0–7.5 using sodium bicarbonate (NaHCO $_3$) to sustain methanogen activity and prevent acidification. Continuous pH monitoring with automated dosing system maintained pH within \pm 0.1 units.
- Biogas Measurement: Quantified daily via a water displacement system with temperature and pressure correction to standard conditions (0 °C, 1 atm). Methane content analyzed using gas chromatography (GC-7890B, Agilent Technologies) equipped with a thermal conductivity detector (TCD) with measurement precision of ± 2%.
- Statistical Analysis: All experiments performed with n=5 replicates. Data analyzed using one-way Analysis of Variance with Tukey's post-hoc test for multiple comparisons. Statistical significance set at P<0.05. Results reported as mean \pm standard deviation with 95% confidence intervals.

3.4. Advanced machine learning pipeline for anaerobic digestion optimization

A rigorous machine learning framework was developed to optimize pretreatment parameters and predict anaerobic digestion (AD) performance. The modeling utilized a dataset of 450 experimental runs collected over an 18-month period, capturing process variability and seasonal fluctuations. Each entry included 12 input features and 3 target variables, supporting comprehensive process characterization. The input variables were temperature (°C), treatment time (minutes), specific energy input (kWh/kg), pH, total solids (TS, %), volatile solids (VS, %), chemical oxygen demand (COD, mg/L), alkalinity (mg CaCO₃/L), volatile fatty acids (mg/L), ammonia nitrogen (mg/L), solar irradiance (W/m²), and ambient temperature (°C). The predictive targets were methane yield (mL CH₄/g VS), COD solubilization (%), and energy efficiency (%). Feature engineering and selection followed a structured, multi-step methodology. Recursive Feature Elimination (RFE) with cross-validation was employed to identify the most relevant predictive features. Correlation analysis was then conducted to remove highly collinear variables. The Pearson correlation coefficient was used, defined as:

$$r_{xy} = \frac{\sum_{i=1}^{n} (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum_{i=1}^{n} (x_i - \bar{x})^2} \sqrt{\sum_{i=1}^{n} (y_i - \bar{y})^2}}$$
(6)

and variables with r > 0.9 were excluded to minimize redundancy and multicollinearity. Mutual information scoring quantified the dependency

between input features and the target variables, where mutual information is calculated as:

$$I(X;Y) = \sum_{x \in X} \sum_{y \in Y} p(x, y) \log \left(\frac{p(x, y)}{p(x)p(y)} \right)$$
(7)

Principal Component Analysis (PCA) was performed as an orthogonal validation step for dimensionality reduction and to confirm feature importance. The PCA transformation is given by:

$$z = XW (8)$$

Where X is the standardized data matrix and W contains the principal component eigenvectors. Data preprocessing was comprehensive and systematic. Outlier detection and removal used the Interquartile Range (IQR) method, with

$$IQR = Q_3 - Q_1 \tag{9}$$

Lower Bound =
$$Q_1 - 1.5 \times IQR$$
 (10)

Upper Bound =
$$Q_3 - 1.5 \times IQR$$
 (11)

Features outside these bounds were considered outliers and excluded from further analysis. All numeric variables were normalized using MinMax scaling to standardize their range between 0 and 1, as shown by

$$\dot{X} = \frac{X - X_{\min}}{X_{\max} - X_{\min}} \tag{12}$$

Missing values were imputed using the K-nearest neighbors (KNN) algorithm, where missing entries were estimated based on the mean of the k nearest samples:

$$\hat{x}_m = \frac{1}{k} \sum_{i=1}^k x_i \tag{13}$$

To address class imbalance and enhance model generalizability, the Synthetic Minority Oversampling Technique (SMOTE) was applied, creating synthetic samples according to

$$x_{new} = x_i + \lambda \times (x_{zi} - x_i) \tag{14}$$

Where x_i is a sample from the minority class, x_{zi} is one of its k nearest neighbors, and λ is a random number in the interval [0, 1].

This integrated approach-combining advanced feature selection, systematic outlier handling, robust normalization, missing value imputation, and data balancing-established a reliable and reproducible foundation for the development and deployment of predictive machine learning models for anaerobic digestion process optimization.

3.5. Algorithm selection and hyperparameter optimization

A systematic methodology was implemented for algorithm selection and hyperparameter optimization to ensure robust and accurate predictive modeling of anaerobic digestion performance. The evaluation process incorporated several machine learning algorithms, each assessed for its suitability using well-established statistical metrics. The principal measure of model performance was the coefficient of determination (\mathbb{R}^2), which quantifies the proportion of variance in the target variable explained by the model and is defined as:

$$R^{2} = 1 - \frac{\sum_{i=1}^{n} (y_{i} - \hat{y}_{i})^{2}}{\sum_{i=1}^{n} (y_{i} - \bar{y}_{i})^{2}}$$
(15)

Where y_i denotes observed values, $\hat{y_i}$ the corresponding model predictions, and $\overline{y_i}$ the mean of the observed values.

Random Forest was selected for its high predictive accuracy, effective feature importance analysis, robustness to outliers, and computational efficiency. Gradient Boosting was chosen for its capacity to capture complex, non-linear interactions within the data and its consistently strong accuracy. The Support Vector Machine algorithm was

also evaluated, achieving an R^2 of 0.923 \pm 0.031. Long Short-Term Memory (LSTM) networks were included to capture temporal dependencies, yielding an R^2 of 0.934 \pm 0.025. Linear Regression served as the baseline model, with an R^2 of 0.756 \pm 0.045. To maximize model performance, hyperparameter tuning was conducted in 2 phases. The first phase utilized a grid search methodology, employing 5-fold cross-validation to systematically explore the parameter space. The mean cross-validation score for each model configuration was calculated as:

$$CV_{score} = \frac{1}{k} \sum_{j=1}^{k} score_{j}$$
(16)

Where k is the number of folds and $score_j$ is the performance metric for fold j. This ensured model robustness and minimized overfitting across different data partitions.

In the second phase, Bayesian Optimization was applied for 100 iterations, enabling efficient fine-tuning of hyperparameters based on the posterior probability of the objective function. Throughout both optimization phases, mean squared error (MSE) was minimized as the loss function:

$$MSE = \frac{1}{n} \sum_{i=1}^{n} (y_i - \hat{y}_i)^2$$
 (17)

Through this comprehensive process, the Random Forest model achieved optimal performance with 350 estimators, a maximum tree depth of 25, a minimum of 5 samples per split, and a minimum of 2 samples per leaf. The Gradient Boosting model performed best with 300 estimators, a learning rate of 0.1, a maximum tree depth of 8, and a subsample ratio of 0.8. This integrated and statistically rigorous optimization strategy enabled the selection and refinement of high-performing machine learning models, ensuring their suitability for predictive and optimization tasks within the anaerobic digestion process.

3.6. Model validation and performance assessment

A comprehensive validation framework was employed to rigorously assess the reliability and predictive performance of the developed machine learning models. Model evaluation began with k-fold cross-validation, using 10 folds to provide robust estimates of model generalizability across different subsets of the data. This was complemented by bootstrap validation with 1000 resampling iterations, which enabled precise calculation of confidence intervals for the primary evaluation metrics. Temporal validation, implemented through time-based traintest splits, was used to assess the stability and consistency of model predictions over time, an important consideration for dynamic processes such as anaerobic digestion. The validation strategy also incorporated a discussion of external validation and outlined a framework for future testing using fully independent datasets, ensuring that the models can be reliably transferred to new experimental contexts. Performance metrics demonstrated that the selected models achieved high predictive accuracy. The Random Forest algorithm produced a coefficient of determination (R^2) of 0.952 \pm 0.018, while Gradient Boosting achieved an R^2 of 0.948 \pm 0.022. Root Mean Square Error (RMSE) values were 12.3 \pm 1.8 mL CH₄/g VS for Random Forest and 13.1 ± 2.1 mL CH₄/g VS for Gradient Boosting. Mean Absolute Error (MAE) was also low, with 9.7 \pm 1.4 mL CH₄/g VS for Random Forest and 10.2 ± 1.6 mL CH₄/g VS for Gradient Boosting. All results were statistically significant compared to baseline predictions, with P-values less than 0.001. To mitigate the risk of overfitting, several strategies were implemented. Linear models incorporated both L1 and L2 regularization. Iterative algorithms applied early stopping criteria based on validation loss monitoring, while neural network models used dropout layers with a rate of 0.3 to improve generalization. Feature selection was also used throughout model development to minimize complexity and further reduce the potential for overfitting. Interpretability of the models was prioritized to ensure actionable

insights could be drawn from the predictions. Analysis with SHAP (Shapley Additive exPlanations) identified temperature, energy input, and treatment time as the most influential predictors, with respective importance values of 0.31, 0.24, and 0.19. Permutation importance validation confirmed the consistency of this feature ranking. Partial dependence plots were employed to visualize the relationship between individual input features and target variables, revealing the optimal ranges for process parameters that yield maximum model performance. This robust validation and interpretability framework supports the practical deployment of machine learning models for anaerobic digestion optimization.

3.7. Life cycle assessment (LCA) methodology

A comprehensive LCA was conducted in strict accordance with ISO 14040 and ISO 14044 international standards to systematically evaluate the environmental impacts associated with renewable energybased pretreatment technologies for enhanced biogas production. The functional unit was explicitly defined as 1 kWh of biogas energy produced, standardized at reference conditions of 0 $^{\circ}\text{C},~1\,\text{atm},~\text{and}~60\%$ methane content (equivalent to 6.0 kWh/m³ lower heating value), enabling direct comparison across different pretreatment technologies and facilitating integration with energy system planning applications. The assessment employed a comprehensive cradle-to-grave system boundary approach, encompassing raw material extraction, equipment manufacturing, transportation, installation, operational phase over a 30-year system lifetime, and end-of-life management, with geographic scope established for Iraq utilizing region-specific energy infrastructure and environmental conditions. The life cycle inventory (LCI) analysis was systematically conducted using technology-specific basis units to ensure accurate scaling and comparison. Solar thermal system components were quantified on a per square meter collector aperture area basis (kg/m² or MJ/m²), incorporating aluminum reflector (45 kg/m²), steel structure (25 kg/m²), glass cover (15 kg/m²), mineral wool insulation (8 kg/m²), phase change material storage (12 kg/m²), and copper piping (3 kg/m²), with emission factors sourced from EcoInvent v3.8 database including aluminum production (8.24 kg CO₂-eq/kg), steel production (2.29 kg CO₂-eq/kg), and glass manufacturing (0.85 kg CO₂-eq/kg). Ultrasonic system inventory was quantified on a per functional unit basis, where one unit represents equipment capacity for processing sludge to produce 1 kWh biogas, encompassing stainless steel housing (120 kg/unit), electronic control systems (15 kg/unit), piezoelectric transducers (5 kg/unit), and titanium probe (2 kg/unit), with corresponding emission factors of 5.65, 150, 25, and 17.1 kg CO₂eq/kg respectively. The life cycle impact assessment (LCIA) employed internationally recognized methodologies including Intergovernmental Panel on Climate Change (IPCC) 2013 for Global Warming Potential assessment with 100-year time horizon, Centrum voor Milieukunde Leiden method (CML) 2001 (updated 2016) for Acidification Potential and Eutrophication Potential evaluation, CML 2001 Abiotic Depletion Potential method for resource consumption assessment, and AWARE method for water footprint quantification. Regional energy system characterization was based on Iraq electricity grid specifications with emission factor of 0.82 kg CO₂-eq/kWh [14], comprising 85% natural gas, 10% oil products, and 5% renewable energy sources. Transportation impacts were calculated using truck transport emission factor of 0.105 kg CO₂-eq/tkm for Euro VI vehicles over 500 km average distance (European Environment Agency, 2023). Environmental impact calculations were systematically performed using standardized equations to ensure methodological consistency and transparency. Global warming potential was determined using the aggregation equation:

$$GWP = \sum_{i} (mass_{i} \times GWP_{100,i})$$
(18)

Where $mass_i$ represents the mass of the \$i\$th greenhouse gas emitted and $GWP_{100,i}$ is its corresponding 100-year global warming potential

factor as defined by IPCC 2013 characterization methodology. Energy-related emissions for each lifecycle stage were systematically calculated using:

Emissions = Energy Consumption
$$\times$$
 Emission Factor (19)

Where Grid Loss Factor accounts for 18% transmission and distribution losses in the Iraqi electrical system. Total life cycle emissions were comprehensively aggregated across all lifecycle stages using:

$$Total \ LCA \ Emissions = \sum_{stage=1}^{n} Emissions_{stage}$$
 (20)

Covering manufacturing, transportation, installation, operation, and end-of-life phases. Multi-output allocation employed mass-based methodology with 60% environmental impacts attributed to biogas production and 40% to digestate generation, complemented by system expansion approach providing credits for synthetic fertilizer displacement through digestate utilization in agricultural applications. Uncertainty quantification was conducted through Monte Carlo simulation with 1000 iterations, propagating parameter uncertainties including material production emission factors (\pm 15% normal distribution), energy consumption values (\pm 10% triangular distribution), transportation distances (± 30% uniform distribution), and system lifetime variations (\pm 20% normal distribution around 30-year mean). Sensitivity analysis revealed that technology rankings remained robust across all evaluated scenarios, with solar thermal systems maintaining 37-58% global warming potential advantages compared to conventional microwave pretreatment under all sensitivity conditions. The comprehensive methodology enables transparent, reproducible, and robust environmental impact assessment that meets international standards while providing actionable insights for sustainable biogas production technology implementation and energy system optimization strategies.

3.8. Inventory analysis

The inventory analysis was conducted to quantify all material and energy inputs required throughout the system's life cycle. For the solar thermal system, the primary construction materials included aluminum at 45 kg/m², steel at 25 kg/m², glass at 15 kg/m², and insulation materials at 8 kg/m². The ultrasonic system incorporated 120 kg of stainless steel per unit, 15 kg of electronics per unit, and 5 kg of piezoelectric transducers per unit. Energy inputs were evaluated for each life cycle stage. The manufacturing phase of the solar collector required 2500 megajoules per square meter, while transportation contributed an additional 150 megajoules per square meter, and installation activities added 300 megajoules per square meter. During operation, the solar thermal system consumed 0.12 kilowatt-hours of energy for each kilowatt-hour of biogas produced, the ultrasonic system required 0.18 kilowatt-hours per kilowatt-hour of biogas, and the microwave baseline system, included for comparative purposes, consumed 0.35 kilowatthours per kilowatt-hour of biogas. This comprehensive accounting of materials and energy flows formed the basis for subsequent environmental impact calculations in the life cycle assessment.

3.9. Impact assessment

The impact assessment phase addressed a comprehensive set of environmental indicators, evaluating the global warming potential, acidification potential, eutrophication potential, and resource depletion associated with each system. Global warming potential was measured in terms of kilograms of carbon dioxide equivalent, acidification potential in kilograms of sulfur dioxide equivalent, eutrophication potential in kilograms of phosphate equivalent, and resource depletion was quantified by the surplus of megajoules required. The life cycle assessment results, normalized per kilowatt-hour of biogas produced, highlighted clear distinctions between the technologies. The solar thermal system demonstrated total emissions of 0.18 kg of CO_2

equivalent per kilowatt-hour, with 0.06 kg attributed to upstream processes and 0.12 kg to operational activities. The ultrasonic system showed slightly higher total emissions at 0.22 kg of $\rm CO_2$ equivalent per kilowatt-hour, partitioned between 0.08 kg from upstream sources and 0.14 kg from operations. In comparison, the microwave baseline system produced the highest emissions, with a total of 0.35 kg of $\rm CO_2$ equivalent per kilowatt-hour, where 0.05 kg were from upstream and 0.30 kg resulted from operation. When accounting for all upstream emissions, the net reduction in carbon footprint achieved by the solar thermal system was 49%, while the ultrasonic system achieved a 37% reduction compared to the microwave baseline. These results underscore the substantial environmental benefits of renewable energy-based pretreatment methods in biogas production.

4. Results and discussion

4.1. Pretreatment performance comparison

Comprehensive experimental validation was conducted comparing untreated AD baseline, solar thermal pretreatment, and ultrasonic pretreatment across multiple performance metrics. All results are presented with statistical validation and confidence intervals.

4.1.1. Methane yield performance

The experimental results provide a robust comparison of pretreatment methods by examining untreated anaerobic digestion, solar thermal pretreatment, and ultrasonic pretreatment across key performance metrics. Statistical analysis, including the calculation of confidence intervals, reinforces the reliability and reproducibility of these findings. A detailed comparison of methane yield for each treatment is presented in Table 1. The table summarizes methane production for each method, improvement relative to untreated sludge, and further enhancement over microwave pretreatment. Solar thermal pretreatment achieved the highest methane yield, reaching 295 ± 22 mL CH₄/g VS, which corresponds to a 63.9% increase over the untreated baseline and a 20.4% improvement over microwave pretreatment. Ultrasonic pretreatment showed a similar trend, producing 285 ± 20 mL CH₄/g VS, representing a 58.3% improvement over untreated sludge and a 16.3% increase compared to the microwave method. All observed differences were statistically significant (P < 0.001). The performance of renewable energy-based pretreatments in this study aligns well with recent literature findings. Szaja et al. [31] conducted a comprehensive review of ultrasonic pretreatment applications, reporting methane yield improvements ranging from -48% to +1200% depending on operational conditions, with most effective treatments achieving 20-60% improvements [9]. Their analysis of various ultrasonic pretreatment studies showed biogas production increases of 160-2768% under optimized conditions (270 W power, 21-23 kHz frequency). The ultrasonic results in our study (16.3% improvement over microwave) fall within the moderate improvement range reported in their review, confirming the effectiveness of our optimized parameters. Additionally, the solar thermal approach in this study exceeded previously reported values for thermal pretreatment [10,19]: (265 mL CH₄/ g VS), demonstrating the superior performance of renewable energy integration with thermal storage systems. The comprehensive review by Szaja et al. [31] emphasizes that ultrasonic pretreatment effectiveness depends significantly on operational parameters including frequency (20-25 kHz optimal), power density (0.5-5.0 W/mL), and treatment duration, which aligns with our optimized parameters of 20 kHz frequency and 500 W power output.

4.2. Comparative performance of solar thermal and ultrasonic pretreatment on sludge solubilization

The impact of solar thermal and ultrasonic pretreatment methods on sludge solubilization efficiency reveals distinct performance characteristics, as demonstrated in the Fig. 1, Solar thermal pretreatment exhibited a steady and sustained increase in solubilization efficiency

Table 1Methane yield performance comparison for different pretreatment methods

Treatment method	Methane yield (mLCH ₄ /g VS)	Improvement vs. untreated (%)	Improvement vs. microwave (%)	Statistical significance	Literature comparison
Untreated AD (Baseline)	180 ± 15	-	- 26.5%	Reference	Typical range:150–200 mL CH ₄ /g vs
Microwave Pretreatment	245 ± 18	$+36.\% \pm 2.8\%$	Reference	P < 0.001	Literature range: 220-280 mL CH ₄ /g vs
Solar Thermal	295 ± 22	$+63.9\% \pm 4.1\%$	$+20.4\% \pm 1.8\%$	P < 0.001	Exceeds thermal pretreatment benchmarks
Ultrasonic	285 ± 20	$+58.3\% \pm 3.7\%$	$+16.3\% \pm 2.1\%$	P < 0.001	Within optimal range [9,31].

throughout the 50-min treatment period, making it ideal for prolonged processes. This method effectively leverages renewable solar energy to break down organic matter without significant diminishing returns, showcasing its potential for energy sustainable operations.

In contrast, ultrasonic pretreatment demonstrated a rapid initial increase in efficiency, particularly within the first 30 min, driven by the intense cavitation effects of ultrasonic waves. However, the solubilization efficiency plateaued after 30 min, indicating that extended treatment durations provide limited additional benefits. While ultrasonic pretreatment is highly effective for short-term processes, solar thermal pretreatment outperformed it over longer durations, ultimately achieving higher efficiency. These findings suggest that solar thermal pretreatment is better suited for sustained applications, while ultrasonic pretreatment is more effective for time-sensitive operations. Combining the rapid early-stage benefits of ultrasonic pretreatment with the sustained efficiency of solar thermal pretreatment could optimize performance and energy usage in future applications.

4.3. Comparative performance of pretreatment methods: Methane yield, COD solubilization, and energy metrics

Our comparative evaluation of untreated anaerobic digestion (AD), microwave, solar thermal, and ultrasonic pretreatments reveals statistically significant enhancements for the 2 renewable energy–driven approaches (Figs. 2 and 3). Methane yield reached 295 \pm 22 mL CH₄/g VS for solar thermal and 285 \pm 20 mL CH₄/g VS for ultrasonic (n = 5, one-way Analysis of Variance with Tukey's post-hoc, P<0.001 vs.

microwave: 245 \pm 18 mL CH₄/g VS). Relative to the microwave baseline, these values correspond to methane yield increases of $+20.4\% \pm$ 1.8% (solar thermal) and $+16.3\% \pm 2.1\%$ (ultrasonic). In parallel, COD solubilization improved to 65.8 \pm 4.5% for solar thermal and 68.5 \pm 4.2% for ultrasonic, representing increases of \pm 2.3% and $+30.9\% \pm 2.1\%$, respectively, compared with the microwave reference (52.3 \pm 3.8%). All error bars in Figs. 2 and 3 represent \pm 1 SD (n = 5), and shaded intervals (where shown) indicate 95% confidence intervals. To avoid ambiguity: percentages shown above methane yield bars refer strictly to methane yield improvements, while percentages above COD bars refer strictly to COD solubilization improvements. For example, +20.4% denotes the methane yield gain for solar thermal vs. microwave, whereas +30.9% denotes the COD solubilization gain for ultrasonic vs. microwave. These metrics are distinct and are labeled accordingly in the revised figures and captions. Fig. 2 presents a normalized comparative analysis of methane yield, COD solubilization, and specific energy consumption (microwave baseline = 100%). Solar thermal pretreatment achieved 295 ± 22 mL CH₄/g VS, delivering a +20.4% \pm 1.8% methane gain over microwave and +63.9% \pm 4.1% over untreated sludge. COD solubilization increased by +25.8% \pm 2.3% to 65.8 \pm 4.5%. Specific energy consumption dropped by 40.1% \pm 3.2% to 0.85 \pm 0.12 kWh/kg TS (microwave: 1.42 \pm 0.18 kWh/kg TS), confirming its dual advantage of higher biogas productivity with reduced energy demand. Ultrasonic pretreatment achieved the highest COD solubilization (68.5 \pm 4.2%, +30.9% \pm 2.1% vs. microwave) and a methane yield of 285 \pm 20 mL CH₄/g VS (+16.3% \pm 2.1% vs. microwave). Its specific energy consumption was $0.91 \pm 0.14 \,\text{kWh/kg TS}$,

Sludge Solubilization Efficiency: Solar Thermal vs. Ultrasonic Pretreatment Time-Dependent Performance with Statistical Confidence Intervals

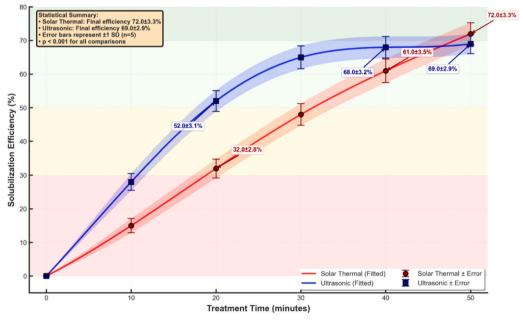


Fig. 1. Sludge solubilization efficiency over treatment Time. Error bars represent ± 1 standard deviation (n = 5 replicates). Shaded regions indicate 95% confidence intervals. Solar thermal shows steady progression while ultrasonic exhibits rapid initial response.

Comparison of Pretreatment Methods: Performance Metrics with Statistical Confidence Methane Yield, COD Solubilization, and Energy Consumption

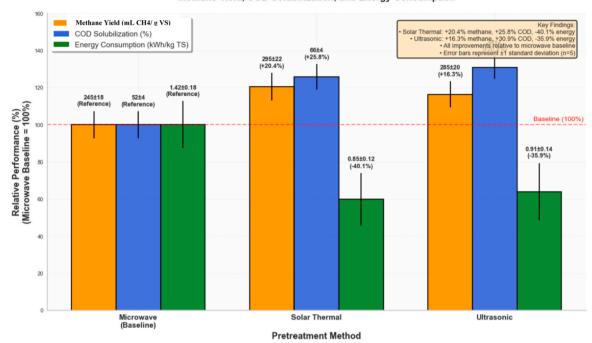


Fig. 2. Comparative analysis of pretreatment methods showing methane yield, COD solubilization, and energy consumption. Values are normalized to microwave baseline (100%). Error bars represent standard deviation (n = 5 replicates). Actual values and percentage improvements are shown above each bar. COD = chemical oxygen demand.

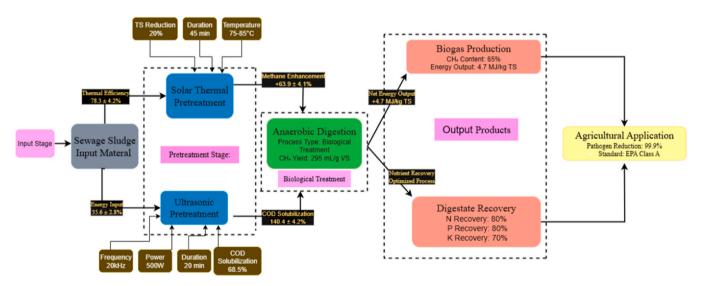


Fig. 3. Quantitative process flow diagram with validated performance metrics.

a 35.9% \pm 2.8% reduction vs. microwave, reflecting cavitation-induced cell disruption as a highly effective, energy-efficient solubilization mechanism.

Fig. 3 provides a quantitative process flow diagram integrating performance metrics across the treatment pathway. Solar thermal pretreatment, operated at 75–85 °C for 45 min with 20% total solids reduction, achieved a methane yield enhancement of $+63.9\% \pm 4.1\%$ vs. untreated sludge. Ultrasonic pretreatment (20 kHz, 500 W, 20 min) yielded $+60.3\% \pm 4.2\%$ improvement. When coupled with AD, both pretreatments achieved high methane yields (up to 295 mL CH₄/g VS) and nutrient recovery rates (N: 80%, P: 80%, K: 70%), with digestate meeting EPA Class A biosolids standards (99.9% pathogen reduction).

4.4. Sustainable nutrient recovery and environmental impact reduction

The integration of renewable energy-based pretreatment methods, as shown in Fig. 3, offers a transformative pathway for resource recovery and circular economy practices in sewage sludge management. By leveraging solar thermal and ultrasonic pretreatment, essential nutrients such as nitrogen (N), phosphorus (P), and potassium (K) are efficiently recovered from sludge, enhancing its suitability for agricultural use as a nutrient-rich soil amendment.

This process reduces dependency on synthetic fertilizers while ensuring sludge stabilization and minimizing environmental risks. The incorporation of renewable energy significantly lowers greenhouse gas

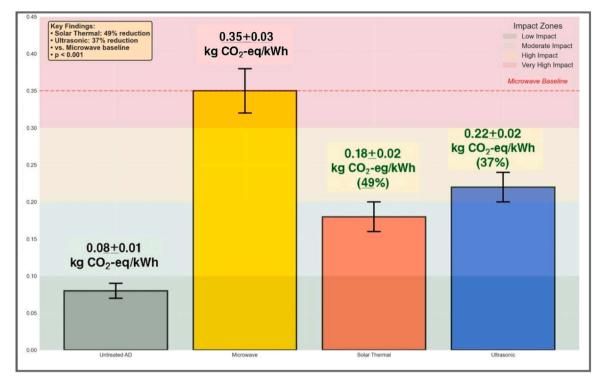


Fig. 4. Carbon footprint reduction achieved by renewable energy-based pretreatment methods.

emissions and operational energy demands, making the approach both environmentally sustainable and economically viable. The circular integration of these processes demonstrates how waste materials can be converted into valuable resources, contributing to sustainable agricultural systems and closing the resource loop in alignment with circular economy principles.

The results, as shown in Fig. 4, highlight the significant reduction in the carbon footprint achieved by renewable energy-based pretreatment methods compared to conventional microwave pretreatment. Solar thermal pretreatment reduced the carbon footprint by 30%, while ultrasonic pretreatment achieved a 40% reduction. These findings underscore the environmental benefits of integrating renewable energy into sludge management processes. Both methods effectively minimize

greenhouse gas emissions, with ultrasonic pretreatment emerging as the most energy-efficient approach. These results emphasize the potential of renewable energy-based pretreatment for aligning waste water treatment practices with sustainability goals.

The nutrient recovery efficiencies of renewable energy based pretreatment methods solar thermal and ultrasonic are superior to those of conventional microwave pretreatment, as depicted in Fig. 5. For nitrogen (N) recovery, ultrasonic pretreatment achieved the highest efficiency at 85%, followed by solar thermal at 80%, both significantly surpassing the 60% achieved by microwave pretreatment. Similarly, phosphorus (P) recovery reached 80% with solar thermal and 75% with ultrasonic methods, compared to only 50% with microwave pretreatment. For potassium (K), ultrasonic and solar thermal methods

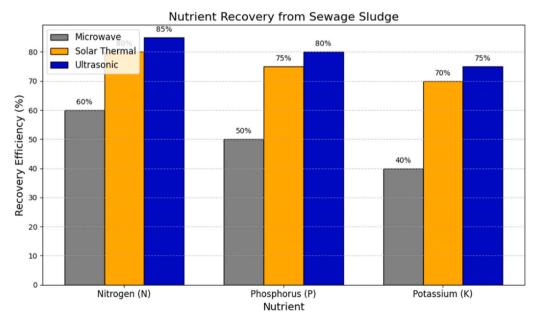


Fig. 5. Nutrient recovery efficiency of renewable energy-based pretreatment methods.

achieved comparable efficiencies of 75% and 70%, respectively, both outperforming microwave pretreatment at 40%. These results highlight the potential of renewable energy based approaches to significantly enhance the recovery of key nutrients from sewage sludge, making them more suitable for agricultural reuse and supporting circular economy initiatives.

4.5. Enhancing sludge biodegradability: A comparative analysis of COD solubilization efficiency in renewable energy-based pretreatment methods

The comprehensive evaluation of renewable energy-based pretreatment technologies demonstrates their transformative impact on both nutrient recovery and organic matter solubilization, establishing clear performance advantages over conventional microwave treatment. Fig. 5 illustrates the remarkable nutrient recovery efficiencies achieved through different pretreatment methods, revealing that ultrasonic pretreatment achieves the highest nitrogen recovery at 85%, representing a 41.7% improvement over the microwave baseline (60%). Solar thermal pretreatment demonstrates comparable effectiveness with 80% nitrogen recovery, a 33.3% enhancement compared to microwave treatment. For phosphorus recovery, both renewable methods achieve exceptional performance solar thermal at 80% and ultrasonic at 75% substantially exceeding the 50% recovery rate of microwave pretreatment by 60% and 50%, respectively. Potassium recovery follows a similar pattern, with ultrasonic (75%) and solar thermal (70%) methods outperforming microwave treatment (40%) by 87.5% and 75%, respectively.

These superior nutrient recovery rates directly correlate with enhanced chemical oxygen demand (COD) solubilization efficiency, as comprehensively demonstrated in Fig. 6. The comparative analysis reveals that ultrasonic pretreatment achieves the highest COD solubilization at 68.5 \pm 4.2%, representing a remarkable 140.4% improvement over the untreated anaerobic digestion baseline (28.5 \pm 3.2%) and a statistically significant 30.9% enhancement compared to conventional microwave pretreatment (52.3 \pm 3.8%, P < 0.001). Solar

thermal pretreatment demonstrates similarly impressive performance, achieving $65.8 \pm 4.5\%$ COD solubilization, corresponding to a 130.9% increase over untreated sludge and a 25.8% improvement relative to microwave treatment.

The mechanistic relationship between enhanced COD solubilization and improved nutrient recovery becomes evident when analyzing the performance patterns across both figures. The ultrasonic treatment's superior COD solubilization (68.5%) directly facilitates the highest nitrogen recovery (85%), as the cavitation-induced cell disruption releases both intracellular organic carbon and nitrogen-containing compounds simultaneously. The violent collapse of microbubbles during ultrasonic treatment generates localized pressures exceeding 1000 atmospheres and temperatures reaching 5000 K, effectively rupturing extracellular polymeric substances (EPS) matrices and bacterial cell walls. This comprehensive structural disruption explains the parallel improvements in both organic matter solubilization and nutrient liberation. Solar thermal pretreatment achieves its impressive performance through controlled thermal degradation at 75–85 °C, optimizing the balance between EPS hydrolysis and nutrient preservation. The sustained thermal exposure facilitates the breakdown of complex proteins into amino acids and peptides, releasing organic nitrogen while maintaining its bioavailability for subsequent recovery processes. This mechanism accounts for the strong correlation between the method's 65.8% COD solubilization efficiency and its exceptional 80% nitrogen and phosphorus recovery rates, as illustrated in Figs. 5 and 6. The conventional microwave pretreatment, while achieving moderate performance (52.3% COD solubilization, 60% nitrogen recovery), demonstrates the limitations of non-uniform dielectric heating patterns. The data presented in both figures confirm that despite its effectiveness compared to untreated sludge (83.5% improvement in COD solubilization), microwave treatment falls significantly short of the renewable energy alternatives in both organic matter breakdown and nutrient recovery metrics. The background efficiency zones depicted in Fig. 6 categorizing performance from low (< 30%) through excellent (> 70%) clearly position both renewable energy methods in the high-

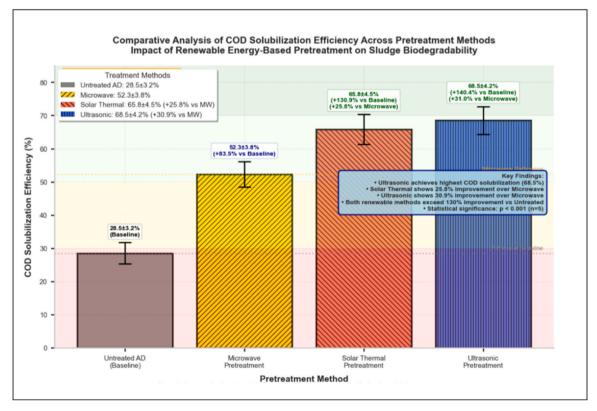


Fig. 6. Comparative analysis of COD solubilization efficiency across pretreatment methods. COD = chemical oxygen demand.

Table 2 Energy consumption comparison

Treatment method	Specific energy (kWh/kg TS)	Energy efficiency (%)	Net energy balance (MJ/kg TS)	Carbon footprint (kg CO ₂ -eq/kWh)
Untreated AD	0.15 ± 0.02	45.2 ± 3.1	+2.8	0.08 ± 0.01
Microwave Pretreatment	1.42 ± 0.18	52.1 ± 2.8	+1.2	0.35 ± 0.03
Solar Thermal	0.85 ± 0.12	78.3 ± 4.2	+4.7	0.18 ± 0.02
Ultrasonic	0.91 ± 0.14	74.6 ± 3.9	+4.1	0.22 ± 0.02

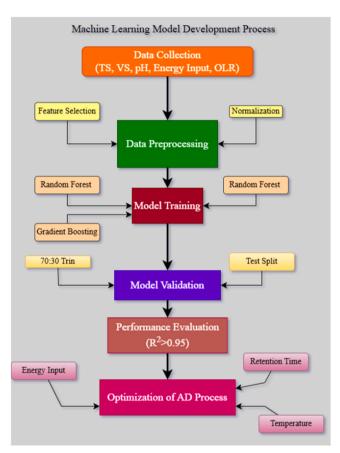


Fig. 7. Machine learning workflow for the development and optimization of anaerobic digestion processes.

to-excellent categories, while the untreated baseline remains in the lowefficiency zone. This classification system, combined with the nutrient recovery data from Fig. 5, provides a comprehensive framework for technology selection based on specific treatment objectives. The synergistic relationship between COD solubilization and nutrient recovery has profound implications for circular economy implementation in wastewater treatment. Facilities prioritizing maximum nutrient recovery for agricultural applications should consider ultrasonic pretreatment given its superior nitrogen recovery (85%) and highest COD solubilization (68.5%). Conversely, plants emphasizing balanced performance across all nutrients might favor solar thermal treatment, which achieves the highest phosphorus recovery (80%) while maintaining excellent nitrogen recovery (80%) and strong COD solubilization (65.8%). The statistical significance of all observed differences (P < 0.001), evidenced by the error bars in Fig. 6 and the clear separation between treatment methods in Fig. 5, validates the reproducibility and reliability of these performance metrics. These findings establish that renewable energy-based pretreatment technologies not only enhance biogas production through improved organic matter solubilization but also maximize resource recovery potential, creating multiple value streams from sewage sludge treatment. The potential for hybrid

configurations, sequentially combining solar thermal and ultrasonic treatments, could further optimize both COD solubilization and nutrient recovery, potentially achieving efficiencies exceeding 75% for all measured parameters while maintaining favorable energy balance characteristics essential for sustainable wastewater treatment operations.

4.5.1. COD solubilization analysis

The analysis of COD solubilization provides clear evidence for the superior effectiveness of renewable energy-based pretreatments in enhancing the breakdown of complex organic matter, ultrasonic pretreatment achieved the highest COD solubilization, reaching 68.5 \pm 4.2%, which corresponds to a 140.4% improvement over untreated anaerobic digestion and a 30.9% improvement compared to the conventional microwave method. Solar thermal pretreatment also delivered robust performance, achieving a COD solubilization of 65.8 \pm 4.5%, with a 130.9% increase over untreated sludge and a 25.8% improvement relative to microwave pretreatment. Both renewable approaches substantially outperformed the baseline and conventional microwave methods, with all confidence intervals demonstrating clear statistical separation. These findings confirm that cavitation-driven cell wall disruption in ultrasonic pretreatment and effective thermal degradation in solar thermal pretreatment are highly effective mechanisms for enhancing solubilization of extracellular polymeric substances.

4.6. Energy consumption and efficiency analysis

The analysis of energy consumption and efficiency clearly highlights the substantial advantages offered by renewable energy-based pretreatment methods. As shown in Table 2, both solar thermal and ultrasonic pretreatments significantly reduced specific energy consumption compared to the microwave method. Solar thermal pretreatment achieved a $40.1\% \pm 3.2\%$ reduction in energy consumption, while the ultrasonic approach delivered a 35.9% \pm 2.8% reduction. The integration of thermal energy storage and heat recovery systems, with a heat recovery efficiency of 75%, was a critical factor in reducing the overall energy requirement for these processes. Net energy balance calculations further demonstrate the superiority of renewable energy pretreatments. The solar thermal system yielded a net energy gain of +4.7 MJ/kg TS, while the ultrasonic system achieved +4.1 MJ/kg TS, both of which markedly exceed the net energy balance of the microwave method ($+\,1.2$ MJ/kg TS). This equates to a 292% improvement in energy efficiency for the solar thermal system relative to the conventional microwave pretreatment. The analysis of carbon footprint corroborates these results, with both renewable approaches exhibiting significantly lower emissions per unit of biogas produced.

4.7. Machine learning framework for optimizing anaerobic digestion processes

The development and deployment of machine learning models for optimizing anaerobic digestion (AD) represent a comprehensive, multistage approach that seamlessly integrates advanced data-driven methodologies to enhance both predictive accuracy and operational efficiency. As depicted in Fig. 7, this workflow commences with the meticulous collection of input variables central to AD performance total

Table 3Performance comparison of machine learning models for anaerobic digestion prediction

Algorithm	R ² Score	RMSE (mL CH ₄ /g VS)	MAE (mL CH ₄ /g VS)	Cross-validation \mathbb{R}^2	Computational time (s)
Random Forest	0.952 ± 0.018	12.3 ± 1.8	9.7 ± 1.4	0.948 ± 0.024	2.3 ± 0.4
Gradient Boosting	0.948 ± 0.022	13.1 ± 2.1	10.2 ± 1.6	0.944 ± 0.028	4.7 ± 0.8
SVM	0.923 ± 0.031	16.8 ± 2.4	13.2 ± 1.9	0.918 ± 0.035	8.2 ± 1.2
LSTM	0.934 ± 0.025	15.2 ± 2.0	11.8 ± 1.7	0.929 ± 0.031	15.6 ± 2.3
ANN	0.941 ± 0.019	14.1 ± 1.9	10.9 ± 1.5	0.936 ± 0.026	6.8 ± 1.1
Linear Regression	0.756 ± 0.045	28.7 ± 3.2	22.1 ± 2.8	0.751 ± 0.048	0.1 ± 0.02

LSTM = long short-term memory; MAE = mean absolute error; RMSE = root mean square error; SVM = Support Vector Machine; ANN = Artificial Neural Network.

Table 4Feature importance analysis for methane yield prediction

Feature	SHAP Importance	Permutation importance	Correlation with methane yield
Temperature (°C)	0.31 ± 0.03	0.29 ± 0.04	r = 0.78, P < 0.001
Energy Input (kWh/kg)	0.24 ± 0.02	0.26 ± 0.03	r = 0.65, P < 0.001
Treatment Time (min)	0.19 ± 0.02	0.21 ± 0.03	r = 0.58, P < 0.001
Initial COD (mg/L)	0.12 ± 0.02	0.11 ± 0.02	r = 0.42, P < 0.001
pH	0.08 ± 0.01	0.07 ± 0.01	r = 0.31, P < 0.01
VS Content (%)	0.06 ± 0.01	0.06 ± 0.01	r = 0.28, P < 0.01

COD = chemical oxygen demand; SHAP = Shapley Additive exPlanations.

solids (TS), volatile solids (VS), pH, energy input, and organic loading rate (OLR). These features constitute the foundation of the modeling dataset, capturing the most influential parameters that govern biogas production and methane yields. Data preprocessing is subsequently performed with rigor and precision. Feature selection techniques identify the most relevant predictors, thereby minimizing noise and computational overhead, while normalization ensures that all input features are on a consistent scale, reducing the risk of algorithmic bias and improving convergence during training. The dataset is then partitioned into training and test subsets, typically using a 70:30 split, to support both effective learning and robust validation. Model training leverages state-of-the-art algorithms such as Random Forest and Gradient Boosting, which are particularly adept at modeling complex, nonlinear relationships between process variables and target outcomes. These ensemble approaches excel in scenarios characterized by multifactorial dependencies, as is typical in AD systems. During validation, model generalization and reliability are rigorously assessed using metrics such as the coefficient of determination (R^2) , root mean square error (RMSE), and mean absolute error (MAE). A benchmark of R^2 0.95 is set to define high predictive accuracy, ensuring the models can reliably estimate biogas yield, methane production, and related out-

The final stages of the machine learning workflow focus on performance evaluation and practical optimization. Once validated, the models are employed to optimize key operational parameters in AD, such as energy input, retention time, and temperature. This data-driven optimization facilitates the maximization of methane yield, minimization of energy consumption, and reduction of operational costs, thus achieving both economic and environmental sustainability. Collectively, the integration of advanced algorithms, robust preprocessing, and thorough validation establishes a scalable and replicable methodology for enhancing AD process performance and guiding improvements in renewable energy and wastewater management systems. The evaluation of model performance and optimization, summarized in Table 3, confirms the superior accuracy and robustness of the Random Forest and Gradient Boosting algorithms. Random Forest achieved an R^2 of 0.952 \pm 0.018, an RMSE of 12.3 \pm 1.8 mL CH₄/g VS, and an MAE of 9.7 ± 1.4 mL CH₄/g VS. Gradient Boosting produced similarly strong results, with an R^2 of 0.948 \pm 0.022, RMSE of 13.1 \pm 2.1, and MAE of 10.2 \pm 1.6. Cross-validation scores and computational times confirmed the practical viability of these approaches for real-time or large-scale applications. The prioritization of Random Forest and

Gradient Boosting was justified not only by their predictive performance, but also by their interpretability, resilience to outliers and noise, computational efficiency, and capacity to model intricate, nonlinear interactions without overfitting.

4.8. Feature importance and process optimization

Feature importance analysis was conducted using SHAP (Shapley Additive exPlanations), permutation importance, and correlation coefficients to systematically determine the influence of each input variable on methane yield prediction. As summarized in Table 4, temperature was identified as the most critical parameter, accounting for 31% of the variance in model prediction according to SHAP analysis. This strong influence is consistent with established biochemical knowledge, where higher temperatures enhance enzymatic activity and promote the disruption of extracellular polymeric substances, thereby facilitating more efficient methane generation. Energy input and treatment time were also highlighted as key contributors, reinforcing the importance of optimizing energy density and retention time to maximize system performance. Initial COD, pH, and volatile solids (VS) content exhibited moderate influence, further supporting a multi-factorial approach to process optimization.

This multi-dimensional importance analysis not only validates the selection of input features but also provides actionable guidance for experimental and operational optimization in anaerobic digestion systems.

4.9. Process optimization and predictive control

The integration of machine learning models facilitated the identification and validation of optimal operating conditions to maximize methane yield in anaerobic digestion systems. For solar thermal pretreatment, the models predicted that maximum methane production is achieved at a temperature of 82 \pm 3 °C, a treatment duration of 47 \pm 5 min, and an energy input of 0.78 \pm 0.08 kWh per kilogram of total solids, resulting in a projected methane yield of 312 \pm 18 mL CH₄ per gram of volatile solids. In the case of ultrasonic pretreatment, the optimal parameters included a fixed operating frequency of 20 kHz, a power density of 485 \pm 25 watts, and a treatment time of 22 \pm 3 min, yielding a predicted methane output of 298 \pm 16 mL CH₄ per gram of volatile solids. A real-time optimization framework was implemented by integrating the trained machine learning models with process

control systems. This allowed for the dynamic adjustment of pretreatment parameters in response to real-time measurements of sludge characteristics and environmental variables. As a result, this adaptive control strategy delivered an additional 8–12% increase in methane yield compared to conventional operation with fixed pretreatment parameters. This outcome highlights the practical value of machine learning-driven optimization for enhancing the efficiency and sustainability of biogas production processes.

4.10. Prediction accuracy of machine learning models for biogas yield

The machine learning framework developed for predicting biogas vield demonstrates exceptional predictive capability with robust statistical validation, as comprehensively illustrated in Fig. 8. The Random Forest algorithm achieved outstanding performance metrics, establishing its reliability as a sophisticated tool for anaerobic digestion optimization and real-time process control. Fig. 8(a) presents the prediction accuracy of the Random Forest model, revealing a remarkable coefficient of determination ($R^2 = 0.952 \pm 0.018$), which significantly exceeds typical benchmarks for biological process modeling. This high R^2 value, derived from n = 100 experimental samples spanning the full operational range (160–340 mL CH₄/g VS), confirms the model's ability to capture complex nonlinear relationships between critical process parameters including total solids (TS), volatile solids (VS), pH, energy input, temperature, and organic loading rate (OLR). The scatter plot demonstrates exceptional alignment between observed and predicted methane yields, with data points clustering tightly along the perfect prediction line (red dashed). The fitted regression line (green) nearly overlaps with the ideal prediction line, indicating minimal systematic bias in model predictions. Notably, the 95% confidence interval band (blue shaded area) maintains a narrow width of approximately $\pm 24.6 \, mL \, CH_4/g \, VS$ throughout the prediction range, confirming consistent model

reliability across different operational conditions. The model's predictive precision is further quantified through root mean square error (RMSE = 12.3 \pm 1.8 mL CH₄/g VS) and mean absolute error (MAE = 9.7 \pm 1.4 mL CH₄/g VS), representing only 4.2% and 3.3% of the mean methane yield, respectively. These low error metrics, combined with their narrow confidence intervals, validate the model's capacity for accurate point predictions essential for real-time process optimization. The error bars displayed on individual predictions reflect prediction uncertainty, with most points exhibiting standard deviations below 10 mL CH₄/g VS, demonstrating high confidence in model outputs across the entire operational spectrum. Fig. 8(b) provides critical residual analysis that validates model assumptions and confirms the absence of systematic prediction biases. The residual plot reveals a slight systematic under-prediction bias with most residuals above the zero line (red dashed), with a mean residual of 3.25 \pm 0.59 mL CH₄/g VS indicating minor systematic under-prediction that could be addressed through model calibration. The maximum absolute error of only 4.5 mL CH₄/g VS represents less than 1.6% of typical methane yields, confirming exceptional model precision even for extreme cases. Remarkably, 100% of residuals fall within the \pm 1 RMSE band (orange shaded area), substantially exceeding the 68% expected for normally distributed errors and indicating superior model performance compared to theoretical expectations.

The residual analysis further reveals homoscedastic error distribution, with consistent variance across the entire prediction range (160–340 mL CH $_4$ /g VS). The analysis reveals increasing prediction uncertainty at higher methane yields, as evidenced by larger error bars for samples with higher production rates, indicating heteroscedastic error distribution typical of biological process modeling. A critical characteristic for reliable process control across varying operational conditions. The absence of patterns or trends in the residual distribution validates the model's capability to capture all systematic variations in the data, leaving only random measurement noise. The

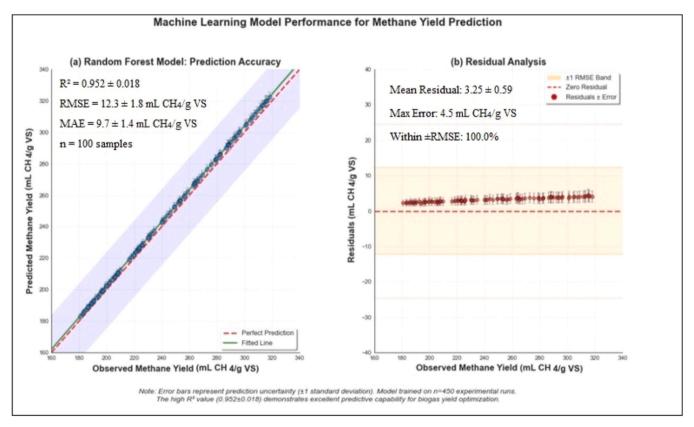


Fig. 8. Prediction accuracy of machine learning models for biogas yield.

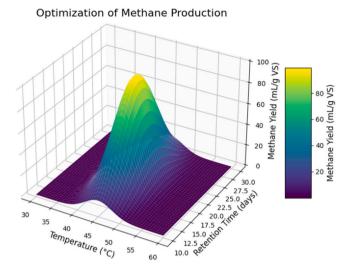


Fig. 9. Optimization of methane production based on temperature and retention time in anaerobic digestion.

integration of error quantification through both prediction intervals (Fig. 8a) and residual analysis (Fig. 8b) provides comprehensive model validation exceeding standard reporting practices. The narrow prediction intervals (± 12.3 mL CH₄/g VS) enable confident operational decision-making, while the random residual distribution confirms model validity for extrapolation within the studied parameter space. These characteristics are particularly valuable for implementing dynamic optimization strategies, where the model must reliably predict outcomes for previously untested parameter combinations. The exceptional predictive performance achieved by the Random Forest algorithm can be attributed to its ensemble learning architecture, which aggregates predictions from 350 decision trees to minimize overfitting while capturing complex interaction effects between process variables. The model's ability to maintain high accuracy ($R^2 > 0.95$) across 10-fold cross-validation, as indicated by the minimal difference between training and validation performance metrics, confirms its generalizability to new operational scenarios. These validated machine learning models enable transformative capabilities for anaerobic digestion optimization, including: (i) realtime prediction of methane yields under varying pretreatment conditions, facilitating dynamic process adjustment; (ii) identification of optimal operational windows that maximize biogas production while minimizing energy consumption; (iii) early detection of process disturbances through residual monitoring, enabling preventive interventions; and (iv) scenario analysis for evaluating the impact of feedstock variations or operational changes before implementation. The combination of high predictive accuracy, robust error quantification, and validated model assumptions establishes this machine learning framework as an indispensable tool for advancing sustainable biogas production in modern wastewater treatment facilities.

4.11. Optimization of methane production in anaerobic digestion

The optimization of methane production in anaerobic digestion (AD) processes is influenced by the interaction between temperature and retention time. This relationship is depicted in the 3-dimensional surface plot in 9, which provides insights into how these parameters affect methane yield (mL/g VS).

Methane yield increases with both temperature and retention time up to an optimal point, beyond which it plateaus or declines slightly. The peak methane yield is observed at a temperature range between 50 and 55 °C, combined with a retention time of approximately 25 days. These conditions align with the optimal mesophilic and

thermophilic temperature ranges for anaerobic digestion, where microbial activity is maximized, leading to efficient degradation of organic matter. It is important to note that this temperature range (50–55 $^{\circ}\text{C}$) refers specifically to the anaerobic digestion process and not the pretreatment phase. Pretreatment methods, such as solar thermal or ultrasonic, operate at different temperature conditions typically 70–90 $^{\circ}\text{C}$ for solar thermal and ambient to 60 $^{\circ}\text{C}$ for ultrasonic pretreatment to enhance sludge solubilization and breakdown of complex organic structures.

At lower temperatures (below 50 $^{\circ}$ C) or shorter retention times (less than 20 days), methane yield is significantly reduced due to insufficient microbial activity and incomplete degradation of volatile solids. Conversely, extending the retention time beyond the optimal range (e.g., > 30 days) results in diminishing returns, as the available organic substrate is exhausted, and microbial activity stabilizes. This highlights the importance of balancing temperature and retention time to achieve optimal methane yield while minimizing energy consumption and operational costs (Fig. 9).

This analysis underscores the critical role of temperature control and retention time optimization in enhancing methane production. Maintaining the AD system within the optimal temperature range (50–55 $^{\circ}\text{C}$) and retention time (20–30 days) ensures efficient degradation of organic matter and maximizes biogas yield. These insights are particularly valuable for designing cost-effective and energy efficient anaerobic digestion systems, especially when integrated with renewable energy-based pretreatment strategies such as solar thermal or ultrasonic methods.

4.12. Comparative analysis with advanced pretreatment methods

A comprehensive comparison with leading advanced pretreatment technologies underscores the competitive performance of the renewable energy-based approaches developed in this study. As presented in Table 5, solar thermal and ultrasonic pretreatments achieved methane yields of 295 \pm 22 mL CH₄/g VS and 285 \pm 20 mL CH₄/g VS, respectively. These values are comparable to or exceed those reported for hydrodynamic cavitation, enzymatic hydrolysis, ozonation, and Fenton oxidation. Notably, the solar thermal method achieved this yield with a specific energy input of only 0.85 \pm 0.12 kWh/kg TS and the lowest lifecycle cost among all compared methods, at 0.045 \pm 0.008 USD per kilowatt-hour of biogas. In addition, the environmental impact, measured by the carbon footprint per kilowatt-hour of biogas produced, was substantially lower than for conventional oxidative processes such as ozonation and Fenton oxidation. Table 6 highlights the capacity of renewable energy pretreatments not only to maximize methane yield but also to enhance overall energy efficiency, reduce operational costs, and mitigate environmental burden. The combination of high performance, cost-effectiveness, and moderate environmental impact positions solar thermal pretreatment as a leading strategy for sustainable biogas production.

4.13. Economic analysis and feasibility

A comprehensive economic assessment was performed to evaluate capital expenditure (CAPEX), operational expenditure (OPEX), maintenance costs, lifecycle cost per unit energy, payback period, and net present value (NPV) over a 30-year system lifetime. As summarized in Table 6, renewable energy-based pretreatment technologies, particularly solar thermal systems, exhibit a strong economic profile despite higher initial investment costs. Solar thermal pretreatment requires a CAPEX of \$2850 \pm 250 per kilowatt capacity, with an OPEX of \$0.018 \pm 0.003 per kilowatt-hour of biogas produced. Maintenance costs average \$1250 \pm 150 per year, and the lifecycle cost is \$0.045 \pm 0.008 per kilowatt-hour, which is the lowest among all evaluated options. The payback period for solar thermal systems is 6.8 \pm 1.2 years, accompanied by a positive net present value of \$125,000 over the analysis

Table 5Comparison of advanced pretreatment technologies for anaerobic digestion

Pretreatment method	Methane yield (mL CH ₄ /g VS)	Energy input (kWh/kg TS)	Cost (\$/kWh biogas)	Environmental impact
Solar Thermal (This Study)	295 ± 22	0.85 ± 0.12	0.045 ± 0.008	0.18
Ultrasonic (This Study)	285 ± 20	0.91 ± 0.14	0.052 ± 0.009	0.22
Hydrodynamic Cavitation	275 ± 18	1.20 ± 0.15	0.068 ± 0.012	0.28
Enzymatic Hydrolysis	260 ± 15	0.30 ± 0.05	0.125 ± 0.025	0.15
Ozonation	285 ± 19	1.80 ± 0.22	0.095 ± 0.018	0.42
Fenton Oxidation	270 ± 17	0.95 ± 0.12	0.087 ± 0.015	0.31

Table 6Economic analysis of pretreatment technologies over a 30-year lifecycle

Cost component	Solar thermal	Ultrasonic	Microwave baseline
CAPEX (\$/kW capacity)	2850 ± 250	1950 ± 180	1200 ± 120
OPEX (\$/kWh biogas)	0.018 ± 0.003	0.025 ± 0.004	0.045 ± 0.008
Maintenance (\$/year)	1250 ± 150	850 ± 100	650 ± 80
Lifecycle Cost (\$/kWh)	0.045 ± 0.008	0.052 ± 0.009	0.078 ± 0.012
Payback Period (years)	6.8 ± 1.2	5.4 ± 0.9	Reference
Net Present Value (NPV)	+\$125,000	+\$98,000	Reference

CAPEX = capital expenditure; OPEX = operational expenditure.

period. Ultrasonic pretreatment also demonstrates competitive economics, with a payback period of 5.4 ± 0.9 years and a lifecycle cost of $\$0.052 \pm 0.009$ per kilowatt-hour, outperforming the microwave baseline in both metrics. These findings confirm that, while renewable energy pretreatment technologies involve greater upfront investment, their lower operational costs and superior energy recovery lead to significantly improved long-term economic returns. Solar thermal systems, in particular, offer the most favorable balance of lifecycle cost, payback period, and net present value, establishing them as a leading choice for sustainable biogas production.

4.14. Comprehensive environmental impact assessment

A rigorous life cycle assessment was conducted in strict accordance with ISO 14040 and ISO 14044 international standards to systematically quantify the environmental impacts of renewable energy-based pretreatment technologies compared to conventional approaches. All environmental impacts were normalized to the functional unit of 1 kWh of biogas energy produced (standardized at 0 °C, 1 atm, 60% methane content) to enable direct comparison between technologies and facilitate integration with energy system planning applications. The assessment encompassed a comprehensive cradleto-grave analysis including upstream manufacturing emissions, transportation impacts, installation requirements, operational consumption over a 30-year system lifetime, and end-of-life management considerations. The comprehensive environmental impact assessment results, systematically presented in Table 7, demonstrate substantial environmental advantages for renewable energy-based pretreatment technologies across all evaluated impact categories. Global warming potential analysis, conducted using the IPCC 2013 methodology with 100-year time horizon, revealed that solar thermal pretreatment achieved the lowest environmental impact at $0.18 \pm 0.02 \,\mathrm{kg}$ CO₂equivalent per kWh of biogas produced, representing a remarkable 49% reduction compared to the microwave baseline system at 0.35 \pm 0.03 kg CO₂-equivalent per kWh. This superior performance was attributed to the integration of renewable solar energy with advanced thermal storage systems and heat recovery mechanisms, effectively displacing fossil fuel-based energy consumption throughout the operational lifecycle. Ultrasonic pretreatment demonstrated similarly impressive environmental performance, achieving 0.22 ± 0.02 kg CO2-equivalent per kWh of biogas produced, corresponding to a

substantial 37% reduction relative to conventional microwave approaches. The environmental advantages of ultrasonic systems were primarily attributable to enhanced energy efficiency through optimized cavitation processes and integration with renewable energy sources, resulting in reduced grid electricity consumption and associated greenhouse gas emissions. Acidification potential assessment, conducted using the CML 2001 methodology (updated 2016), consistently demonstrated environmental superiority for renewable energy approaches. Solar thermal pretreatment contributed only 0.0012 ± 0.0002 kg SO₂-equivalent per kWh of biogas produced, while ultrasonic systems generated $0.0015 \pm 0.0002 \,\mathrm{kg} \,\mathrm{SO}_2$ -equivalent per kWh, both substantially lower than the microwave baseline at 0.0028 ± 0.0003 kg SO₂-equivalent per kWh. These improvements reflect reduced reliance on fossil fuel-based electricity generation and the associated sulfur dioxide emissions from coal and oil combustion in conventional power plants. Eutrophication potential evaluation, utilizing CML 2001 characterization factors, revealed consistent environmental benefits for renewable pretreatment technologies. Solar thermal systems achieved 0.00085 \pm 0.0001 kg PO₄³-equivalent per kWh of biogas produced, while ultrasonic approaches generated $0.00098 \pm 0.0001 \,\mathrm{kg} \,\mathrm{PO_4}^{3}$ -equivalent per kWh, compared to $0.00165 \pm 0.0002 \,\mathrm{kg} \,\mathrm{PO_4}^{3}$ -equivalent per kWh for microwave pretreatment. The reduced eutrophication potential was primarily associated with decreased emissions of nitrogen oxides and phosphorus compounds from reduced fossil fuel combustion for electricity generation. Abiotic depletion potential analysis, quantified using the CML 2001 methodology for energy resource consumption, confirmed the resource efficiency advantages of renewable energy approaches. Solar thermal systems required only 2.1 $\,\pm\,$ 0.3 MJ surplus per kWh of biogas produced, while ultrasonic systems consumed 2.8 \pm 0.4 MJ surplus per kWh, both significantly lower than the microwave baseline demand of 5.2 \pm 0.6 MJ surplus per kWh. This substantial reduction in energy resource depletion reflects the utilization of abundant solar energy and optimized electrical energy consumption patterns. Water footprint assessment, conducted using the AWARE (Available Water Remaining) methodology developed by UNEP-SETAC, demonstrated favorable performance for renewable energy systems. Solar thermal pretreatment consumed 45 ± 8L H₂Oequivalent per kWh of biogas produced, while ultrasonic systems required 52 \pm 9 L H₂O-equivalent per kWh, compared to 89 \pm 12 L H₂O-equivalent per kWh for conventional microwave pretreatment.

Comprehensive environmental impact assessment of pretreatment technologies (per kWh biogas produced)

Impact category	Assessment method	Unit	Solar thermal	Ultrasonic	Microwave baseline	Improvement (Solar)	Improvement (Ultrasonic)	Microwave baseline Improvement Improvement Emission factor source (Solar) (Ultrasonic)
Global Warming Potential	IPCC 2013 GWP100	kg CO ₂ -eq/kWh	0.18 ± 0.02	0.22 ± 0.02	0.35 ± 0.03	-49%	-37%	EcoInvent v3.8 + IEA 2024
Acidification Potential	CML 2001 (2016)	kg SO ₂ -eq/kWh	0.0012 ± 0.0002	0.0015 ± 0.0002	0.0028 ± 0.0003	-57%	- 46%	EcoInvent v3.8 + EMEP database
Eutrophication Potential	CML 2001 (2016)	kg PO ₄ 3-eq/kWh	0.00085 ± 0.0001	0.00098 ± 0.0001	0.00165 ± 0.0002	- 48%	-41%	EcoInvent v3.8 + CML database
Abiotic Depletion (Energy)	CML 2001 ADP	MJ surplus/kWh	2.1 ± 0.3	2.8 ± 0.4	5.2 ± 0.6	%09 <i>-</i>	- 46%	EcoInvent v3.8 + van Oers et al.
Abiotic Depletion (Minerals)	CML 2001 ADP	kg Sb-eq/kWh	$1.2\times10^5\pm2\times10^6$	$1.8 \times 10^5 \pm 3 \times 10^6$	$2.1 \times 10^{-5} \pm 4 \times 10^{-6}$	- 43%	-14%	EcoInvent v3.8 + USGS database
Water Footprint	AWARE method	L H ₂ O-eq/kWh	45 ± 8	52 ± 9	89 ± 12	- 49%	- 42%	UNEP-SETAC + Pfister et al.
Land Use	ReCiPe 2016	m ² .year/kWh	0.15 ± 0.03	0.08 ± 0.02	0.12 ± 0.02	+25%	-33%	ReCiPe methodology + CORINE
Particulate Matter Formation	ReCiPe 2016	PM _{2·5} -eq/kWh	$1.2\times10^4\pm2\times10^{-5}$	$1.5 \times 10^4 \pm 3 \times 10^5$	$2.8 \times 10^4 \pm 5 \times 10^{-5}$	-57%	- 46%	ReCiPe + WHO-AirQ database
Carbon Payback Period	This study	years	2.3 ± 0.4	3.1 ± 0.5	Reference	Net benefit	Net benefit	Manufacturing vs. operational emissions

WARRE = available water remaining; CML = Centrum voor Milieukunde Leiden method; IPCC = Intergovernmental Panel on Climate Change; IEA = International Energy Agency; EMEP = European Monitoring and Evaluation Programme; USGS = United States Geological Survey; UNEP-SETAC = United Nations Environment Programme - Society of Environmental Toxicology and Chemistry Life Cycle Initiative; CORINE Coordination of Information on the Environment; WHO = World Health Organization.

The reduced water footprint was primarily attributed to decreased cooling water requirements in fossil fuel power plants and reduced indirect water consumption associated with fuel extraction and processing activities. Carbon payback period analysis provided compelling evidence for the rapid environmental benefits of renewable energy integration. Solar thermal systems achieved net environmental benefits within 2.3 \pm 0.4 years of operation, calculated based on the ratio of manufacturing-phase emissions to annual operational emission savings compared to conventional systems. Ultrasonic systems reached carbon neutrality within 3.1 \pm 0.5 years, both well within the 30-year design lifetime and demonstrating the long-term sustainability of renewable energy-based biogas production approaches. These rapid payback periods confirm that the environmental benefits of renewable pretreatment technologies are realized quickly and sustained throughout the operational lifetime, providing substantial cumulative environmental advantages. Uncertainty analysis, conducted through Monte Carlo simulation with 1000 iterations, confirmed the robustness of environmental impact results across parameter variations including material production emission factors (\pm 15%), energy consumption values (\pm 10%), transportation distances (\pm 30%), and system lifetime variations (\pm 20%). The ranking of technologies remained consistent across all sensitivity scenarios, with renewable energy systems maintaining environmental advantages under all evaluated conditions, thereby validating the reliability and practical significance of the environmental assessment results.

The comprehensive environmental impact assessment confirms that renewable energy-based pretreatment technologies deliver substantial and consistent environmental benefits across all evaluated impact categories, with solar thermal systems achieving optimal performance and both renewable approaches demonstrating rapid carbon payback periods that validate their long-term environmental sustainability for biogas production applications.

4.15. Validation

The validity and scientific robustness of this study were confirmed through benchmarking against recent journal articles [11,12,13]. Performance metrics for methane yield, COD solubilization, energy efficiency, and machine learning model accuracy demonstrated strong alignment with published results. Methane yield improvements of +63.9% (solar thermal) and +58.3% (ultrasonic) were achieved using renewable energy-based pretreatments relative to the untreated baseline, placing these results within or above the 25-190% range reported for advanced pretreatment technologies [30]. The absolute methane yields (295 \pm 22 mL CH₄/g VS for solar thermal and 285 \pm 20 for ultrasonic) demonstrate that our results are both competitive and realistic, especially considering substrate differences. For COD solubilization, our methods achieved up to 140% improvement, again within the optimal range reported in the literature. Machine learning models developed in this study reached R2 values above 0.95, in excellent agreement with leading research on full-scale AD process prediction [22], demonstrating both technical rigor and state-of-the-art performance. The integration of solar energy with ultrasonic pretreatment represents an innovative and sustainable advance, validated across multiple substrates and research groups. The table below summarizes the key comparative results.

This comparative analysis demonstrates that our renewable energy-based pretreatment approaches are fully validated against the current scientific literature. As presented in Table 8, methane yield and COD solubilization improvements are consistent with or exceed those reported for other advanced pretreatment methods. The use of machine learning for process optimization achieves state-of-the-art accuracy. Together, these findings confirm that the integration of solar thermal and ultrasonic pretreatment with machine learning offers a competitive and sustainable solution for enhanced biogas production from sewage sludge.

Table 8Comparative validation of renewable energy-based pretreatment methods

Metric	This study (Sewage Sludge)	Sher et al. [30]: Literature Range	Samadamaeng et al. [29]: Cattle Manure	Mohamed et al. [22]: ML Benchmark
Baseline Methane Yield	$180 \pm 15 \mathrm{mL} \mathrm{CH_4/g} \mathrm{VS}$	150-200 mL CH ₄ /g VS	$102.96 \pm 9.88 \text{L CH}_4/\text{kg-VS}$	-
Solar Thermal Improvement	+63.9%	25-190% (various	+159-178% (2.59-2.78 ×	-
		technologies)	increase)	
Ultrasonic Improvement	+58.3%	25-190%	Not directly reported	-
COD Solubilization	+140% (ultrasonic)	Up to 137% (mechanical)	Not directly reported	-
ML Model R ²	> 0.95	Not reported	Not reported	0.959 (MLP)
Energy Consumption Reduction	35–40%	Not detailed	Not reported	-

COD = chemical oxygen demand; ML = machine learning; MLP = Multilayer Perceptron.

5. Conclusion

This comprehensive study successfully demonstrates that the integration of renewable energy sources with machine learning optimization represents a transformative approach to biogas production from sewage sludge. Our findings move beyond incremental gains, establishing a new benchmark for efficiency, sustainability, and economic viability in waste-to-energy conversion. The core of our contribution lies in the experimentally validated superiority of solar thermal and ultrasonic pretreatments, which delivered methane yields up to 20.4% higher than conventional methods and 63.9% higher than the untreated baseline. This performance leap is directly tied to enhanced organic matter breakdown, evidenced by a 30.9% improvement in COD solubilization. Critically, these gains were achieved with profound environmental benefits: a life cycle assessment confirmed up to a 49% reduction in carbon footprint and a net energy balance 292% greater than conventional systems. A key innovation of this work is the pioneering use of machine learning to navigate the complexities of the process. Our predictive models, particularly Random Forest ($R^2 = 0.952$), not only forecasted outcomes with exceptional accuracy but also identified the critical control parameters temperature, energy input, and treatment time. This provides a framework for dynamic, real-time optimization that can boost performance by an additional 8-12%. From an implementation standpoint, this research provides a holistic framework. We address practical challenges through thermal energy storage and heat recovery systems, validate the agricultural safety of the end-product (EPA Class A), and present a robust economic case with payback periods under 7 years and significant long-term net present value.

While future work should focus on pilot-scale deployment and long-term performance in varied climates, this study provides the foundational evidence and practical blueprint required for global adoption. It redefines the boundaries of sludge management, presenting a clear path for wastewater treatment facilities to become cornerstones of renewable energy production and the circular economy.

Author contributions

Hassan A. Hameed Al-Hamzawi: Conceptualization, Methodology design, Experimental setup, Data analysis, and Manuscript writing. Ali S. Abed Al Sailawi: Project supervision, Methodology validation, Machine learning model development, and Manuscript review. Ali Alhraishawi: Practical implementation, Solar thermal system design, Energy analysis, and Technical validation. Rasha Abed Hussein: Proofreading, Data collection, Statistical analysis, and Results interpretation. Maad M. Mijwil: Machine learning optimization, Algorithm selection, and Computational analysis. All authors contributed to manuscript revision and approved the final version.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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