



Chemical Pretreatment of Spent Coffee Grounds for Biomethane Production: A Review of Biochemical Methane Potential Assays

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Abstract

Spent coffee grounds (SCG) constitute a rapidly growing organic waste stream with a complex lignocellulosic matrix that restricts microbial degradation during anaerobic digestion (AD). This review evaluates the effects of oxidative, alkaline, and acidic pretreatments on SCG solubilization and biomethanation performance. The methodology involved a comparative synthesis of reported batch mesophilic biochemical methane potential (BMP) assays employing distinct substrate-conditioning strategies. Particular attention was given to operational conditions, soluble chemical oxygen demand (SCOD), biogas yield, and methane yield. The synthesized evidence indicates that ambient alkaline treatment using 8% w/w NaOH produced a robust methane yield of 392 mL CH₄/g VS. In comparison, severe thermo-acidic treatment with 1.5% w/w H₂SO₄ at 121°C generated 381.12 mL CH₄/g VS. Treating SCG with 4% w/w H₂O₂ for 24 hours at room temperature increased SCOD by 713.02% relative to untreated SCG, indicating substantial solubilization of previously inaccessible organic matter. This condition produced 642.69 mL biogas/g VS and 345.53 mL CH₄/g VS without elevated temperature or pressure. Overall, pretreatment effectiveness cannot be assessed solely from solubilization rates; chemical dosage, energy use, and inhibitor formation must also be evaluated. Standardized comparative BMP testing and routine toxicity assessments are essential to forecast digester performance and improve sustainable waste valorization accurately.

Keywords: *Spent Coffee Grounds, Pretreatment, Anaerobic Digestion, Methane Production, Biogas*

INTRODUCTION

Environmental pollution driven by intensive fossil fuel use and poor waste management presents a major challenge to sustainable development, marked primarily by rising carbon emissions and global warming. Consequently, developing renewable energy and sustainably utilizing organic waste are critical to mitigating pollution and advancing a circular economy through resource recovery (Subbarao et al., 2023).

One organic waste that continues to increase is spent coffee grounds (SCG). Around 6-8 million tonnes of SCG are generated annually, and most still end up in landfills or are incinerated, even though these disposal methods risk further pollution, including greenhouse gas emissions (Franca & Oliveira, 2022).

The recalcitrant structure and relatively high lignin and lipid contents of SCG limit microbial degradation during anaerobic digestion. Accordingly, various pretreatment strategies have been investigated to enhance organic matter solubilization and methane recovery. Among these, NaOH-based alkaline pretreatment can improve methane yields while facilitating the generation of value-added products within integrated biorefinery schemes (Ribeiro et al., 2023).

Dilute acid-thermal pretreatment effectively increases the solubility of SCG organic materials

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before digestion (Semaan et al., 2023). Oxidative pretreatment using hydrogen peroxide also increases biogas and methane yield (Sayoud et al., 2025)

Anaerobic Digestion (AD) converts organic matter in biomass waste into renewable biogas. This process uses microorganisms to decompose organic components under oxygen-free conditions, producing a flammable gas mixture (Uddin & Wright, 2023). The generated biogas can be combusted for combined heat and power generation, providing renewable heat and electricity for decentralized applications (Subbarao et al., 2023).

This conversion occurs through four biochemical stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Mokraoui et al., 2023). Biochemical methane potential (BMP) tests are the primary laboratory method for characterizing substrate methane potential and biodegradability, although limited standardization reduces comparability across studies (Filer et al., 2019).

Pretreatment determines how well spent coffee grounds (SCG) digest anaerobically. (Bułkowska, 2025) reviewed SCG pretreatment methods comprehensively, yet few studies compare energy input, chemical cost, and methane yield across chemical methods under standardized conditions.

This review fills that gap by comparing oxidative, alkaline, and acidic pretreatments and their effects on SCG composition, with particular attention to how lignin degradation drives biomethane recovery. We evaluate each method's effectiveness through BMP assay results, biodegradability, and digestion stability.

LITERATURE REVIEW

Physical and Chemical Composition of Spent Coffee Grounds (SCG)

The physicochemical composition of SCG varies significantly with climate, location, coffee species, post-harvest processing, and roasting methods (Sayoud et al., 2025). In general, SCG contains a complex lignocellulose. In terms of the inorganic fraction, or ash content, SCG has a very low ash content, generally below 1% of its dry weight (Franca & Oliveira, 2022).

The roasting process plays an important role in forming melanoidins. Melanoidins are brown polymers that make up about 13-25% of dry weight and are rarely found in green coffee beans, but their amount increases significantly as the roasting rate increases (Iriundo-DeHond et al., 2021).

Based on its chemical composition, SCG is dominated by lignocellulose, proteins, lipids, and various phenolic compounds. In lignocellulose, hemicellulose accounts for about 35 to 39%, cellulose about 9 to 12%, and lignin about 19 to 24% of dry matter (Solomakou et al., 2022). The lipid fraction contains about 7 to 21% (Bevilacqua et al., 2023), while protein accounts for about 13 to 17% of the dry matter (Singh et al., 2023).

This lignocellulose will be used as a renewable energy source, but it also resists microbial degradation (Copik et al., 2023). Cellulose and hemicellulose are potential substrates for the enzymatic hydrolysis process, while lignin still needs to be degraded as it blocks the hydrolysis process (Bułkowska, 2025)

Pretreatment

Pretreatment modifies or removes resistant lignocellulose components, increases solubility, and improves enzyme access to carbohydrates and proteins (Trinh et al., 2022). Reducing lignin levels is the main strategy to achieve effective pretreatment (Ravindran et al., 2017). Studies show that biomass pretreatment can increase biomethane yields by making the substrate easier for microorganisms to degrade (Atelge et al., 2021).

Lignin content can also inhibit methanogenesis, particularly at high concentrations, and lead to the accumulation of volatile fatty acids (VFAs). This indicates that lignin acts not only as a

physical barrier preventing microbial access to cellulose but also inhibits the microbial communities involved in methane formation (Piątek et al., 2021).

Alkali pretreatment swells the lignocellulose wall and causes some lignin to decompose. Furthermore, adding alkalis can help neutralize acidic compounds produced during the degradation process (Giroto et al., 2018). Among chemical agents, NaOH is frequently reported as a highly effective lignin decomposer that can substantially improve methane yields under specific operational conditions (Kang et al., 2018).

Specific evidence from the 8% NaOH pretreatment of SCG in BMP assays showed the best anaerobic digestion performance (392 mL CH₄/kg VS) due to enhanced lignin degradation and the release of dissolved organic carbon (Giroto et al., 2018).

Acid pretreatment can dissolve hemicellulose from the solid phase into liquids and accelerate the dehydration-condensation reaction of sugars that remove lignin, thereby supporting enzymatic performance (Fan et al., 2025). Under efficient conditions, acid pretreatment can achieve near-complete breakdown of hemicellulose molecules (Semaan et al., 2023).

When using acid pretreatment, operating temperature must be controlled because cellulose dissolves above 160°C, which can reduce fermented sugar yield (Martins et al., 2022). Acid-based hydrothermal pretreatment can hydrolyze cellulose and partially hydrolyze lignin and hemicellulose (Semaan et al., 2023).

Oxidative pretreatment with H₂O₂ is employed because of its strong oxidizing capacity, minimal residual formation, and decomposition into environmentally benign water and oxygen with limited by-products. Its primary mechanism involves lignocellulosic delignification, generating reactive species such as hydroxyl and hydroperoxyl radicals that promote organic matter degradation and enhance subsequent AD.

However, H₂O₂ pretreatment may yield lower methane production than alkaline or acid treatments and can oxidize VFAs, which are essential AD intermediates, while potentially promoting ammonia accumulation that inhibits methanogenic activity. (Sayoud et al., 2025).

Biochemical Methane Potential Assays Method

The BMP assay determines the maximum methane yield from an organic substrate under optimal anaerobic conditions (Yasim & Buyong, 2023). The BMP assay measures the maximum methane volume produced per unit of volatile solids (VS) from a substrate anaerobically incubated with an active methanogenic inoculum (Jingura & Kamusoko, 2017).

BMP test durations of 20 to over 100 days limit applicability for time-sensitive operational decisions, though early parameter estimation using first-order kinetic modeling can reduce testing time to as little as 7 days for rapidly biodegradable substrates (Da Silva et al., 2018). Under psychrotrophic farm-scale conditions, BMP assays showed no statistically significant correlation with full-scale digester performance, as field methane yield (0.36 Nm³ CH₄/kg VS) far exceeded batch test values (0.18–0.19 Nm³ CH₄/kg VS) (Madrigal et al., 2026).

RESEARCH METHOD

This review combined a structured search with narrative synthesis to evaluate SCG pretreatments for enhanced biomethane production. Peer-reviewed English-language articles published between 2016 and 2026 were retrieved from ScienceDirect and Scopus. Searches used the Boolean query: ("Spent Coffee Grounds" OR "SCG") AND ("Pretreatment" OR "Delignification") AND ("Biomethane Production" OR "Anaerobic Digestion" OR "Biogas Yield").

After full-text screening, we included studies that investigated physical and chemical pretreatments of SCG and reported quantitative biogas or methane yields. Data were extracted on raw-SCG physicochemical properties, pretreatment conditions, and digestion performance.

Descriptive and comparative analyses were then used to identify overall performance patterns and assess each pretreatment's effectiveness in improving SCG biodegradability.

Collected articles were screened based on specific inclusion criteria: publication in peer-reviewed scientific journals, explicit investigation of chemical pretreatments (specifically oxidative, alkaline, or acidic methods), and the presentation of quantitative biogas or methane yields via BMP assays. Conversely, articles lacking gas production data, focusing on physical or biological pretreatments, or relying solely on simulation without experimental validation were excluded. These data were analyzed descriptively and comparatively to identify trends in pretreatment effectiveness.

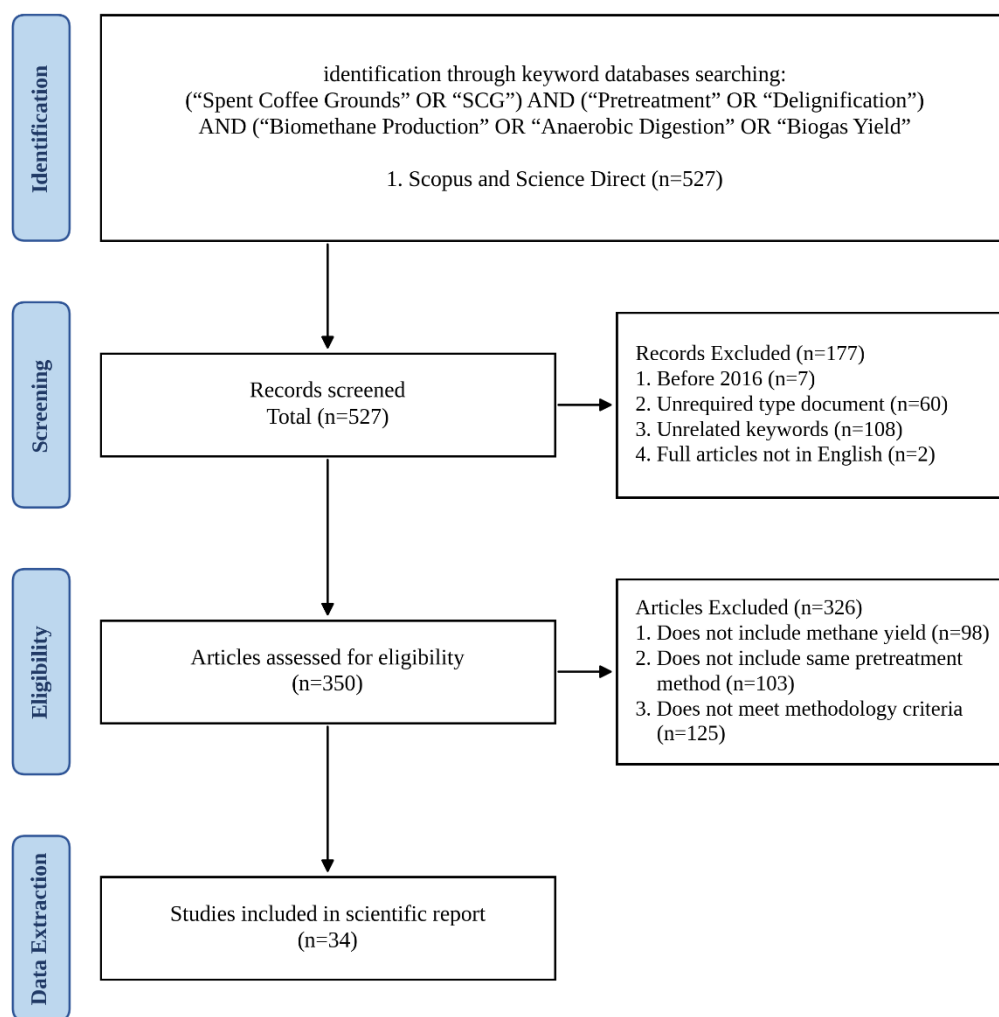


Figure 1. Flow Diagram of Data Extraction

FINDINGS AND DISCUSSION

Oxidative Pretreatment

All oxidative pretreatments tested on SCG raised methane yield above the baseline of 295 mL CH₄/g VS, though the gains varied by strategy. H₂O₂ at 4% w/w for 24 h at room temperature pushed yield to 345.53 mL CH₄/g VS; amine oxide at 73% and 120°C for 1- 5 h reached 337.4 mL CH₄/g VS. H₂O₂ also raised soluble COD by up to 713% and sugar solubilization by up to 818%, pointing to oxidation releasing soluble organics that feed anaerobic digestion (Sayoud et al., 2025).

Amine oxide treatment boosted substrate porosity by 63% and bioavailable sugars by 27%,

improving microbial access to organic matter and likely aiding biodegradation and methane formation (Oliva et al., 2024). However, increased solubilization does not always correspond to higher methane yield; therefore, *soluble chemical oxygen demand* (SCOD) or dissolved sugars are not sufficient as the sole predictor of BMP performance.

Therefore, H₂O₂ should be discussed more appropriately as a promising oxidative option under mild conditions, rather than as an optimal condition, particularly given energy and toxicity considerations. Although higher H₂O₂ concentrations tend to increase SCOD and latent sugars, this cycle is not always followed by increased biogas and methane production, so the initial condition indicator cannot be used alone to predict BMP performance.

Practically, this method is interesting because it can be carried out in the surrounding environment without high temperature or pressure, making it simpler than other pretreatments (Sayoud et al., 2025).

Table 1. Methane Yields from SCG Under Varying Oxidative Pretreatment Conditions

Pretreatment Method	Substrate	Methane Yield	Improvement	Source
Initial conditions	SCG	295 mL CH ₄ /g VS	-	(Sayoud et al., 2025)
2% w/w H ₂ O ₂ , 24 h at room temperature	SCG	263.61 mL CH ₄ /g VS	Low alkalinity & high ammonium inhibit methanogens	(Sayoud et al., 2025)
4% w/w H ₂ O ₂ , 24 h at room temperature	SCG	345.53 mL CH ₄ /g VS	SCOD increased by up to 713%; sugar solubilization up to 818%	(Sayoud et al., 2025)
73% Amine Oxide solution, 120 °C for 1–5 h	SCG	337.4 mL CH ₄ /g VS	Porosity increased by 63%; bioavailable sugars increased by 27%	(Oliva et al., 2024)

Alkaline Pretreatment

NaOH pretreatment disrupts the lignocellulosic matrix of SCG to enhance anaerobic digestion, though excessive sodium loading induces methanogenic toxicity (Giroto et al., 2018). High alkaline loadings also inhibit hydrogen and volatile fatty acid production from SCG slurry at high organic loads due to excessive chemical use (Lawrence et al., 2025). Sodium ion concentrations reaching 2.2 g/L exceed the inhibition threshold for methanogenic archaea, disrupting intracellular ionic balance and ATP formation via osmotic stress (Bułkowska, 2025).

Table 2. Methane Yields from SCG Under Varying Alkaline Pretreatment Conditions

NaOH Pretreatment Condition	Substrate	Methane Yield	Improvement	Source
NaOH 8% w/w, 24h, ambient	SCG	392 mL CH ₄ /g VS	Best AD performance; 24% higher lignin	(Giroto et al., 2018)

			degradation vs. untreated	
NaOH 0.13 M, 70.5°C (thermo-alkaline)	SCG	263.3 mL CH ₄ /g COD	Optimal yield; highest solubilization (36.4%) at 0.18 M did not translate to the highest methane	(Kim et al., 2018)
NaOH: Urea (co-digestion with corn stover)	SCG + Corn Stover	353 mL CH ₄ /g VS	80.25% of theoretical maximum; 88.5% delignification	(Belay et al., 2026)
NaOH 2.0 M, 60°C, 1h (hydrolysis)	SCG + Pulp Wastewater	148.5 g/L VS	Maximized total sugar concentration (181 g/L) for co-digestion	(Shen et al., 2022)

The data indicate that prolonged alkaline exposure without added thermal energy is effective; pretreatment with 8% w/w NaOH for 24 hours under ambient conditions resulted in the best overall AD performance. This method achieved a peak methane yield of 392 mL CH₄/g VS and enabled 24% higher lignin degradation than untreated SCG (Giroto et al., 2018). The dataset highlights a distinct operational trade-off between the physical solubilization of the biomass and ultimate methanogenic efficiency.

When applying a thermo-alkaline pretreatment of 0.13 M NaOH at 70.5°C, researchers achieved an optimal yield of 263.3 mL CH₄/g COD; however, the data note that increasing the chemical severity to 0.18 M to achieve the highest solubilization rate (36.4%) did not translate into the highest methane yield (Kim et al., 2018). A similar disconnect is observed under severe hydrolysis conditions.

Using a highly concentrated 2.0 M NaOH pretreatment at 60°C for 1 hour maximized total sugar concentration to 181 g/L for co-digestion, yet it produced the lowest biogas output in the dataset (115 mL CH₄/g COD) when co-digested with pulp wastewater (Shen et al., 2022). Conversely, synergistic approaches using multi-chemical pretreatment and co-digestion show strong efficacy in bridging the gap between structural degradation and biological conversion.

Applying combined NaOH and urea pretreatment for co-digesting SCG with corn stover achieved a highly efficient 88.5% delignification rate. This structural breakdown translated effectively to biomethanation, yielding 353 mL CH₄/g VS, which accounts for 80.25% of the theoretical maximum yield (Belay et al., 2026)

Acid Pretreatment

Acid pretreatment (diluted or concentrated) optimally triggers hemicellulose hydrolysis to improve carbohydrate degradation, achieving near-perfect saccharification even if crystalline cellulose is only partially dissolved. In contrast, alkaline hydrolysis focuses more on the lignin fraction (Auma Omondi & Aluda Kegode, 2023). Acid pretreatment significantly alters both the volumetric yield and the kinetic efficiency of anaerobic digestion, with outcomes heavily dictated by acid classification, thermal severity, and substrate composition.

Acid application substantially improves bioenergy recovery. Extended pretreatment with 2.5% (v/v) hydrochloric acid (HCl) for 45 minutes produced 119.7 mL of biogas/g VS, a 286% improvement over the control at the same time point (Semaan et al., 2023). Sulfuric acid (H₂SO₄) shows strong efficiency when paired with severe thermal conditions; exposing biomass to 1.5% (w/w) H₂SO₄ at 121 °C for 90 minutes achieves a robust methane yield of 381.12 mL CH₄/g VS, a

116.91% improvement over thermal-only pretreatment frameworks (Gosławski, 2024).

Mechanistically, acid pretreatment targets the hemicellulose fraction and enhances the release of soluble compounds, especially when combined with thermal conditions. Using 1.00 M H_2SO_4 at a milder 60 °C for 1 hour to pretreat SCG in a co-digestion setup with pulp wastewater generates a specific yield of 115 mL CH_4 /g COD, which still represents an eightfold biomethanation enhancement compared with raw pulp wastewater alone (Shen et al., 2022).

Table 3. Methane Yields Across Acid Types and Pretreatment Conditions

Pretreatment Method	Substrate	Methane Yield	Improvement	Source
HCl; 2,5% (v/v); 45 min	SCG	119.7 mL CH_4 /g VS	286% improvement vs. control at the same time point	(Semaan et al., 2023)
H_2SO_4 ; 1.5% (w/w), 121 °C, 90 min	SCG	381.12 mL CH_4 /g VS	116.91% vs. thermal-only	(Gosławski, 2024)
H_2SO_4 ; 1.00 M, 60 °C, 1 h, co-digested with pulp wastewater	SCG + Pulp wastewater	115 mL/g COD	8×raw pulp wastewater or ~700%	(Shen et al., 2022)
Acetic acid; 2.5–10% (v/v); 90°C, 1 h	Coffee Pulp	0.92 L biogas/g VS (79.8% CH_4)	Reduced HRT from 21 to 15 days	(Nava-Valente et al., 2021)

Pretreatment utilizing acetic acid at concentrations of 2.5-10% (v/v) and 90°C for 1 hour achieves a peak volumetric yield of 0.92 L biogas/g VS, characterized by a highly enriched methane fraction of 79.8% (Nava-Valente et al., 2021). Taken together, the data show a clear divergence in operational benefits: while severe thermo-acidic conditions using H_2SO_4 effectively double methane outputs relative to non-chemical thermal baselines, organic agents like acetic acid offer a compelling alternative by maximizing biogas volume and methane concentration while compressing the biological processing timeline.

Table 4. Performance Comparison of Chemical Pretreatment of SCG and Specific Methane Yields.

Pretreatment Method	Optimal Condition	Substrate	Standardized Yield (mL CH_4 /g VS)	Notes	Source
Control	Without pretreatment	SCG	295.00	BMP Baseline	(Sayoud et al., 2025)
Oxidative	4% w/w H_2O_2 , ambient, 24 h	SCGs	345.53	High solubilization, low energy, but BMP evaluation is needed	(Sayoud et al., 2025)

Alkaline	8% w/w NaOH, ambient, 24 h	SCG	392.00	Highest yield among	(Giroto et al., 2018)
Acid	1.5% w/w H ₂ SO ₄ , 121 °C, 90 min	SCG	381.12	Effective, but high severity	(Gosławski, 2024)

Alkaline pretreatment gave the highest standardized methane yield among the strategies compared, reaching 392.00 mL CH₄/g VS with 8% w/w NaOH at ambient conditions for 24 h, followed by acid pretreatment at 381.12 mL CH₄/g VS using 1.5% w/w H₂SO₄ at 121°C for 90 min. In comparison, oxidative treatment with 4% w/w H₂O₂ at ambient conditions for 24 h reached 345.53 mL CH₄/g VS, compared with a control of 295.00 mL CH₄/g VS (Sayoud et al., 2025).

NaOH likely drives the higher yield by breaking down SCG's lignocellulosic structure and exposing more biodegradable matter to hydrolysis and anaerobic digestion (Giroto et al., 2018); acid pretreatment's gain over the control probably reflects enhanced hydrolysis and solubilization of acid-sensitive fractions under heat and acidic conditions (Gosławski, 2024); and H₂O₂ pretreatment also improved biodegradability relative to the control, though it trailed both alkaline and acid treatment (Sayoud et al., 2025)

CONCLUSIONS

The comprehensive evaluation of BMP assays establishes that chemical pretreatments successfully overcome the lignocellulosic recalcitrance of SCG, significantly enhancing organic solubilization and subsequent biomethane production. Among the evaluated strategies, ambient alkaline pretreatment utilizing 8% w/w NaOH for 24 hours delivered the highest standardized performance, achieving a peak methane yield of 392 mL CH₄/g VS and demonstrating a 24% improvement in lignin degradation relative to untreated substrates.

Thermo-acidic conditioning using 1.5% w/w H₂SO₄ at 121 °C for 90 minutes was similarly robust, driving hemicellulose hydrolysis to generate 381.12 mL CH₄/g VS. Alternatively, oxidative conditioning with 4% w/w H₂O₂ at room temperature increased SCOD by up to 713.02%. It yielded 345.53 mL CH₄/g VS without requiring elevated thermal or pressurized inputs.

Optimizing SCG pretreatment is essential for sustainable valorization, supporting circular economy frameworks while mitigating greenhouse gas emissions from inefficient organic waste management. However, inconsistent reporting of inhibitory byproducts across the literature precludes definitive quantitative assessment of methanogenic toxicity.

LIMITATIONS & FURTHER RESEARCH

Experimental conditions and reporting units differ across the primary literature, so direct numerical comparisons carry uncertainty. Several studies used co-digestion frameworks or other lignocellulosic substrates instead of SCG alone, which limits how cleanly SCG-specific effects can be isolated.

This synthesis rests on laboratory-scale BMP assays, and net energy balance remains preliminary. Furthermore, the literature reports inconsistently on inhibitory byproduct formation across studies, limiting a definitive quantitative assessment of methanogenic toxicity. Future research should adopt standardized frameworks such as testing all pretreatments under the same SCG, inoculum, and BMP conditions while rigorously accounting for study quality and publication bias.

Future studies should therefore standardize BMP protocols to facilitate reliable cross-study comparisons and investigate pretreatment conditions that maximize organic matter accessibility

and methane conversion while minimizing substrate losses and microbial inhibition. Standardized BMP procedures, coupled with first-order kinetic modeling, could further enhance data comparability and reduce experimental testing time.

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