

Acid-Base Pretreatment of Sugarcane Bagasse for Anaerobic Digestion into Biogas in the Presence of Cowdung Mesophically to Remove H₂S Through Adsorption on XFe₂O₃-Cement Sand

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The objectives of this investigation were to employ an acid-base pretreatment technique for extracting sugars from bagasse and to explore anaerobic biogas production within a 1-liter biogas digester utilizing cow manure as the substrate, maintaining a C/N ratio of 22–30 at a controlled temperature of 35 °C for a hydraulic retention time (HRT) of 30 days. The study revealed that the acid-base pretreatment was highly effective in disrupting lignin linkages, thereby enhancing cellulose accessibility. This approach also yielded the highest biogas production of 84.84 mL/g after 16 days, compared to only 76.09 mL/g from untreated bagasse. Furthermore, the acid-base pretreated bagasse demonstrated superior biomethane production, achieving approximately 357.33 mL/g of volatile solids (VS), whereas untreated bagasse yielded 325.34 mL/g of VS. The initial pH of the anaerobic digestion process plays a pivotal role in influencing methanogenic activity, which is critical for methane generation. Notably, methanogens present in cow manure were capable of producing methane even under highly acidic conditions over the sixteen-day period. Additionally, the xFe₂O₃/cement-based sand absorbents exhibited a limited hydrogen sulfide (H₂S) absorption capacity of 57.7%, whereas the xFe₂O₃-based sand absorbents achieved a remarkable removal efficiency of 95% to 100% for H₂S. The optimal absorber was identified as cementitious sand with a 4% wt Fe₂O₃ content, effectively eliminating H₂S at gas flow rates between 59 and 89 mL/min. Moreover, the reusability of the Fe₂O₃/cement-based sand could be substantially improved through a washing process followed by exposure to atmospheric oxygen.

Keywords: Biogas, cement-based sand, Ferric oxide, Hydrogen sulfide removal, Sugarcane bagasse.

1. Introduction

The demand for refined petroleum products in Thailand, particularly jet fuel, is projected to escalate in 2024, with an anticipated average growth of 24.2%, reaching 16.8 million liters per day, compared to 13.5 million liters per day in 2023. Similarly, diesel consumption is expected to experience a modest increase of 0.4%, reaching 69.1 million liters per day, while gasoline consumption is forecasted to ascend by 3.7%, culminating at 32.6 million liters per day (Srisatabusaya, 2024). This upward trajectory underscores a consistent amplification in energy utilization. Within the Thai demographic, agricultural activities, particularly sugarcane cultivation, remain predominant. It is forecasted that sugarcane production will attain 105.86 million tons during the 2022-2023 period (S.R. Department, 2023). The residual by-products of sugarcane processing, notably 26.8% bagasse, hold significant potential for biogas generation, thus contributing to a reduction in dependence on gasoline and liquefied petroleum gas (Paulose, 2020; Prasad, 2020).

Several strategies can be implemented to improve the efficiency of utilizing sugarcane bagasse, with anaerobic digestion (AD) being recognized as a promising approach for biogas production (Smith, 2022; Jones & Lee, 2021). Nevertheless, the complex molecular structure of bagasse, which includes hemicelluloses, cellulose, and lignin, presents a significant obstacle for anaerobic digestion. As a result, various pretreatment methods are necessary to enhance microbial degradation (Brown & Davis, 2020; Wilson, 2019).

Biogas production typically takes place in anaerobic conditions, where inoculants such as cow dung or rumen fluid are combined with pre-treated sugarcane bagasse. Several factors influence the efficiency of this process, including operational temperature, pH, C/N ratio, and liquid-to-solid ratio (Brown, 2020). The pre-treatment methods applied and the specific inoculum utilized play a crucial role in the breakdown of lignocellulose and hemicellulose, which is comparable to the processing of sugarcane bagasse. This approach softens the biomass and enhances microbial access to cellulose surfaces, thereby facilitating hydrolysis and augmenting biogas production (Jones & Smith, 2019; Wilson & Lee, 2021). Various pre-treatment techniques for biomass, such as lignocellulose derived from sugarcane bagasse, include physical, biological, and chemical methods, each with its respective benefits and limitations (Smith & Davis, 2021; Brown et al., 2020). According to Abraham et al. (2020), these methods can be employed individually or in combination. Anaerobic digestion (AD) is one such method, utilizing efficient microorganisms to recycle bio-waste under anaerobic conditions. This process involves the enzymatic decomposition of organic materials, converting agricultural waste into valuable resources for microbial use. The decomposition is accelerated, leading to the production of bioactive compounds within a period of two to four weeks. These processes not only reduce greenhouse gas emissions but also avoid the generation of thermal energy, unpleasant odors, and are both cost-effective and efficient.

The anaerobic digestion (AD) process yields biogas, primarily composed of methane (CH_4), carbon dioxide (CO_2), and trace amounts of hydrogen sulfide (H_2S). However, the presence of H_2S poses a significant challenge as it can corrode engine-generator systems, thereby compromising the efficiency of the combustion process (Brown, 2021). Moreover, reducing H_2S concentrations is crucial not only for optimizing biogas energy output but also for safeguarding human health, as prolonged exposure to H_2S can lead to serious health hazards

(Smith & Jones, 2020). Consequently, implementing an effective containment strategy within the anaerobic digestion system to absorb or eliminate H₂S is of paramount importance.

Previous studies have identified numerous methods for the removal of hydrogen sulfide (H₂S), including dissolution in a liquid medium such as water or a caustic solution, or adsorption onto solid substrates like iron oxide (Fe₂O₃) (Lien et al., 2014) or activated carbon (Coppola & Papurello, 2018). Additionally, Mrosso et al. (2020) highlighted the use of red rock, notable for its high iron oxide content, to enhance the efficacy of H₂S removal. Cement-based materials are widely utilized in the construction of biogas infrastructure—such as pre-tanks, digesters, post-digesters, and storage tanks—due to their cost-effectiveness. However, there is a lack of comprehensive data on cement-based sand absorbents that offer water resistance, high-temperature stability, and long-term durability, despite their frequent use in cement-based biogas digestion systems (Voegel et al., 2015). Thus, there remains a pressing need to improve absorbent materials that are both economically viable and easily adaptable for H₂S removal.

2. Materials and Methods

2.1. Preparation and analysis of substrates

2.1.1 Preparation of substrates

The Research and Development division of The Green Millennium Company Limited, located in Prawet, Bangkok, Thailand, provided the sugarcane bagasse used in this study. The bagasse was subjected to dehydration at 55 °C until it reached a consistent mass. Subsequently, it was processed through a laboratory knife mill to standardize its particle size to 1–20 mesh (0.1–2 mm) using a sieve mesh, in preparation for the subsequent chemical pretreatment process.

2.1.2 Analysis of substrates

The comprehensive study was conducted at Mahasarakham University's laboratory, located in Kham Riang Subdistrict, Kanwichai District, Maha Sarakham, Thailand. The analysis of carbon and nitrogen content in the bagasse, both prior to and following its integration with cow dung, was performed utilizing the Perkin Elmer CHNS/O 2400 Analyzer. A substantial body of research underscores the significance of the carbon-nitrogen (C/N) ratio in facilitating biogas generation through anaerobic digestion (Zheng et al., 2014; Maryana et al., 2014; Tanimu et al., 2014). Biomass sources exhibit a wide range of C/N ratios, from 32 to 150:1. Moreover, it has been documented that microorganisms involved in fermentation processes exhibit a significantly higher rate of carbon assimilation compared to nitrogen, with a ratio of 25 to 30:1 (Zheng et al., 2014; Tanimu et al., 2014). According to the findings presented by Kaur et al. (2020), the ideal C/N ratio for optimizing biogas production is 30:1. The unprocessed bagasse exhibited a C/N ratio of 130:1, but the ratio decreased to 29:1 when bagasse was combined with cow dung (Kaur et al., 2020).

2.1.3 Acid-Base Pretreatment

The sugarcane bagasse was subjected to an acid-alkaline pretreatment protocol as follows: Initially, 10 grams of bagasse were suspended in 200 milliliters of a 1% (v/v) sulfuric acid solution. The suspension was agitated at 100 °C for 40 minutes. Post-treatment, the acid-treated bagasse was filtered and thoroughly rinsed with hot water until the wash water achieved

a neutral pH. Subsequently, the neutralized bagasse was treated with 200 milliliters of a 2% (w/v) sodium hydroxide solution, followed by agitation at 100 °C for an additional 40 minutes. Upon completion of the alkaline pretreatment, the bagasse was again rinsed with hot water to neutral pH, followed by a washing step using a 50-mM citrate buffer with a pH of 4.8. Finally, the solid residue was oven-dried at 55 °C until it reached a consistent weight.

2.2 Preparation of $x\text{Fe}_2\text{O}_3$ cement-based sand

In this study, the absorbent material utilized was a cementitious sand composite, formulated by blending sand, a single component of cement (fine binding powder), and water in a weight-to-volume ratio of 2:1:1, respectively. After the mixture was placed into a cylindrical plastic mold, a central cavity measuring 0.45 cm \times 0.45 cm \times 0.15 cm was created. The cementitious sand underwent a curing process for forty-eight hours at ambient temperature. Iron (Fe) was incorporated into the cement-based sand at a mass ratio of 8.25 weight percent. During a one-hour period, the cement-based sand exhibited an absorption capacity of 0.486 FeCl_3 and 3.00 H_2O . Following a twenty-four-hour drying period in a hot air oven, the iron oxide concentration within the sand was determined to be 8.25 percent. The Fe_2O_3 /cement-based sand was subsequently immersed in a 0.36 sodium hydroxide solution containing 3.00 H_2O for twenty-four hours, followed by another drying period at room temperature overnight. The sand was then subjected to calcination at 550 °C for six hours. To reduce the presence of H_2S in biogas, a composite sand formulation of $x\text{Fe}_2\text{O}_3$ /cement was developed, where the weight percentage of x varied between 1 and 41.8. Comparative experiments were conducted to evaluate the performance of $x\text{Fe}_2\text{O}_3$ /cement-based sand against a control group consisting of cement-based sand without FeCl_3 addition.

2.3 Experimental procedures

The study employed a laboratory-scale bio-digester constructed from high-density polyethylene (HDPE) and outfitted with four sharp, razor-like stir sticks. The digester had a fixed-bed anaerobic digestion capacity of 1.0 L. To accommodate the release of gas and mitigate the risk of explosion during periods of excessive or sudden gas production, a plastic pipe was connected to one end of the bio-digester and submerged in a 500-mL beaker partially filled with water. The opposite end was connected to a corrosion-resistant metal pipe fitted with a valve, enabling the gas to pass through an H_2S absorber before being quantified using a gas analyzer. Gas measurements were taken bi-daily over a period of 30 days. Additionally, a stirrer was inserted into the central aperture of the bio-digester to facilitate daily agitation of the slurry (Kulawong et al., 2022).

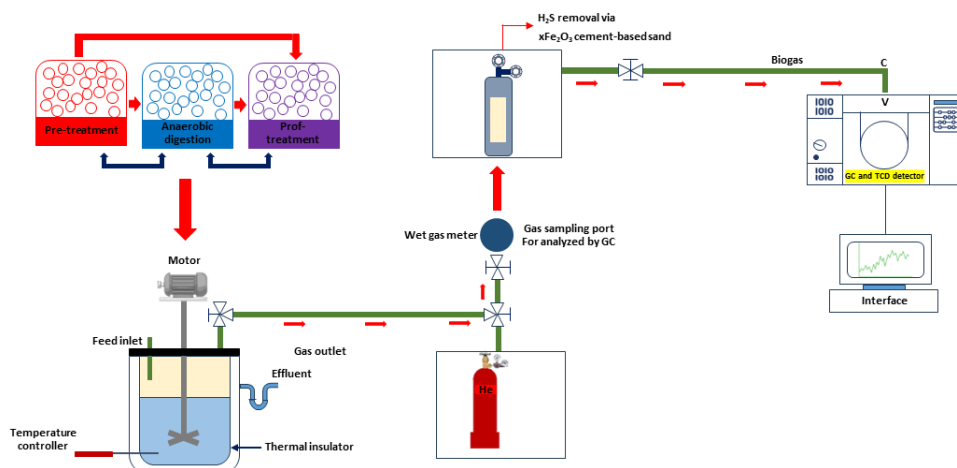


Figure 1. Schematic of the 5-liter bioreactor contained the absorbent material made of $x\text{Fe}_2\text{O}_3$ -cement-based sand to make fermented biogas from treated sugarcane bagasse.

A weighing scale was used to determine the required mass of cow manure and chemically treated bagasse. A glass thermometer with a temperature range of 0–110 °C was used to measure the daily temperature of the slurry. The thermometer was placed into the biodigester through the cork. The digital pH meter was utilized to measure the pH of the slurry. There are two distinct categories of digesters: ADR0, employed for the purpose of regulating anaerobic digestion and only comprising unadulterated bagasse, and ADR1, which encompasses bagasse that has undergone treatment with NaOH. The combination of pretreated and untreated bagasse with cow dung is achieved using a 1:2 ratio, which is subsequently followed by a 1:3 ratio when the resulting mixture is combined with water. To maintain a consistent temperature, the digesters were submerged in a water bath maintained at a temperature of 350 °C. To achieve optimal conditions for ADR1, $x\text{Fe}_2\text{O}_3$ -cement-based sand was supplied to the H_2S removal section in order to purify the produced biogas.

2.4 Chemical Analysis

2.4.1 Biomethane from Anaerobic Digestion

Over a 30-day period, data were systematically gathered from all digesters, with measurements taken every other day. The total biogas volume was measured using a 100-mL syringe connected to an anaerobic biodigester. Quantification of the biogas was conducted utilizing an Agilent Technologies 6890 N gas chromatograph, equipped with dual columns (Porapak and molecular sieve), flame ionization detection (FID), and thermal conductivity detection (TCD). For quantitative calibration, standard gas mixtures were utilized, with helium employed as the carrier gas. A 10- μL biogas sample was injected into the gas chromatography system through the sample port, with the detection and oven temperatures set at 300 °C and 270 °C, respectively. The flow rates for helium, hydrogen, and air were maintained at 26 mL/min and 80 psi, 30 mL/min and 40 psi, and 300 mL/min and 60 psi (Tanimu et al., 2014; Kaur et al., 2020).

The determination of bagasse biodegradability involved a comparison between the theoretical

methane potential and the cumulative methane production observed over the incubation period, as depicted in equation (1).

$$\text{BD\%} = \frac{\text{BMP}_{\text{end}}}{\text{BMP}_{\text{ThOFC}}} \times 100 \quad (1)$$

Where BMP end (mLCH₄/gVS) is the total amount of methane produced till the incubation period end., and BMP_{ThOFC} (mLCH₄/gVS) is the theoretical methane potential.

At regular intervals of two to four days, hydrogen sulfide levels were measured using a portable gas detector alongside a manual gas pump equipped with rapid-response detection tubes (Jentys et al., 1999). Biogas samples were collected in 2-L Tedlar® gas-sampling bags and quantified using a wet-drum gas meter. To purify the biogas, 99.99% helium carrier gas (HE-HP, Oxygen Tech Co., Ltd., Thailand) was utilized to transport the biogas to the fixed-bed H₂S remover packed with xFe₂O₃-cement-based sand at varying flow rates ranging from 59 to 189 mL/minute (see Figure 1). The purified biogas was subsequently analyzed using a gas chromatograph (Agilent Technologies 6890 N) under the specified conditions, and the removal efficiency of H₂S was proportionally converted into purified biogas percentages.

2.4.2 Volatile Fatty Acids from Anaerobic Digestion

The Volatile Fatty Acid (VFA) concentration was assessed utilizing the alkali titration method, wherein the sample was diluted with 10 to 100 mL of distilled water. The diluted sample was then transferred into a 20 mL Erlenmeyer flask using a pipette. To this flask, three drops of a methyl orange indicator solution were added. The mixture was subsequently titrated with 0.1 mL of potassium hydroxide (KOH) until the solution developed a faint pink color. The stoichiometric analysis revealed that the VFA concentration in the sample, expressed in grams per liter, was equivalent to the volume of KOH used during titration (Kaur et al., 2020).

3. Results and Discussion

3.1 Anaerobic digestion of bagasse for biogas production

The daily biogas production at 35 °C, as illustrated in Figure 2, indicates a substantial increase in biogas yield during the first 16 days of Hydraulic Retention Time (HRT). This surge is likely attributable to the rapid decomposition of soluble sugars within the substrate. Following this initial 16-day period, a marked decline in biogas production is observed, likely due to the extensive conversion of organic material into biogas during this retention phase. The study further reveals that the peak biogas output occurs on the sixteenth day, with acid-alkaline treated bagasse yielding 84.84 mL/g of volatile solids (VS), compared to the untreated bagasse, which generates 76.09 mL/g of VS, reflecting a distinct difference in biogas productivity (Kaur et al., 2020).

Multiple factors substantiate this assertion. Firstly, acid-alkaline pretreatment has been identified as the most efficacious approach for disrupting lignin's cross-linking structures (Zheng et al., 2014). Secondly, this pretreatment method has been demonstrated to effectively penetrate cellulose crystals and disrupt the microfibrils within the sample (Kim et al., 2020; Pedrosa et al., 2022). Finally, the alkaline process enhances the hydrolysis of hemicellulose,

thereby enabling microbial enzymes to more effectively access the cellulose within the sample (Yang et al., 2011; Laca et al., 2019). A thorough comprehension of acid treatments is essential to mitigate corrosive impacts and avert potential damage to cellulose during the acid treatment process (Kargarzadeh et al., 2018).

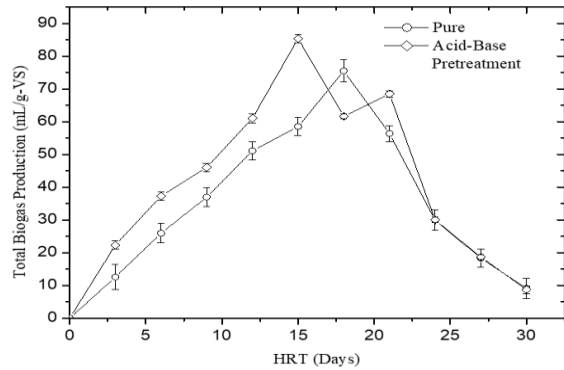


Figure 2. The total amount of biogas produced by the anaerobic digestion of sugarcane bagasse (with and without acid-base pretreatment) in a 1-L bio-digester at a temperature of 30°C and with cow dung added over 30 days.

Figure 3 depicts the cumulative biomethane production over a 30-day HRT period. The experimental results reveal that the acid-alkaline pretreatment of bagasse produces the highest biomethane yield, approximately 357.33 mL/g of VS. Conversely, untreated bagasse generates biogas at a rate of 325.34 mL/g of VS at 35 °C. These findings corroborate the previously stated assertion that acid-alkaline pretreatment is markedly more effective in augmenting biogas production from sugarcane bagasse compared to its untreated form. The efficiency of this pretreatment method lies in its ability to effectively break down the structural linkages within the fibrous matrix, thereby substantially enhancing biomethane yield (Zheng et al., 2014; Srisatabusaya, 2024).

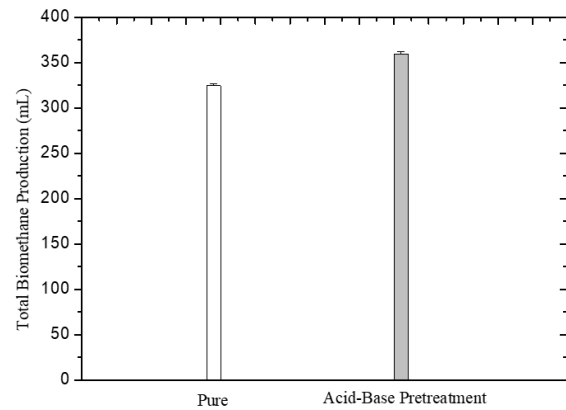


Figure 3. The overall biomethane generation from pure and Acis-Base-pretreated sugarcane bagasse at 35 °C throughout a 30-day period of HRT in the present cow dung

3.2 Effects of pH from VFAs evolved

The effectiveness of methanogens, which are crucial for methane generation, is significantly influenced by the pH levels within anaerobic digestion (AD) processes. The initial pH of the fermentation medium ranged between 6.9 and 7.0. Following a 48-hour period of substrate addition, the pH levels dropped to 6.63 for the untreated process and 5.99 for the acid-alkaline treatment, as depicted in Figure 4. This decline in pH is primarily due to the accumulation of volatile fatty acids (VFAs), which are more prevalent in the acid-alkaline process due to the breakdown of glucose into oligosaccharides, subsequently serving as a more accessible substrate for methanogens, thereby enhancing biomethane production (Kaur et al., 2020; Stanley et al., 2022). In contrast, the pH of the untreated process fluctuated between 6.6 and 7.0, reflecting the challenges in decomposing lignin and hemicellulose into fermentable sugars (Poddar et al., 2022; Abraham et al., 2020). Methanogens remain the optimal microorganisms for efficient biomethane production.

As a result, methanogens originating from cow manure exhibited a remarkable capacity to endure the acidic environment within the anaerobic digestion (AD) system (Mrosso et al., 2020), leading to sustained methane production over a 16-day period. Both treatment methods displayed a significant reduction in pH levels, reflecting enhanced substrate digestibility (Coppola & Papurello, 2018). The alkali titration method confirmed the generation of a substantial quantity of volatile fatty acids (VFAs), correlating with a marked increase in biogas output during the first 16 days. Beyond this period, the pH levels stabilized between 5.27 and 5.68, accompanied by a slight decline in biogas production from day 17 to day 30. This reduction in biogas yield may be attributed to the depletion of critical substrates and the associated pH drop, which signals the terminal phase of certain methanogens' life cycle (Abid et al., 2021; Kaur et al., 2020).

The pH stability during the anaerobic digestion (AD) processes observed in this study aligns with findings from other researchers, contributing to the consistent production of elevated levels of volatile fatty acids (VFAs). Among the prominent VFAs identified are acetic acid, butyric acid, and hexanoic acid (Voegel et al., 2015). As corroborated by the research of Lu et al. (2020), anaerobic digestion conducted under unregulated pH conditions tends to produce higher concentrations of VFAs. A distinct relationship has been established between a pH value of 7 and VFA concentrations ranging from 4.5 to 20 gCOD/L, and from 10 to 45 gCOD/L. Conversely, a pH of 5 is associated with VFA concentrations ranging between 4 to 18 gCOD.

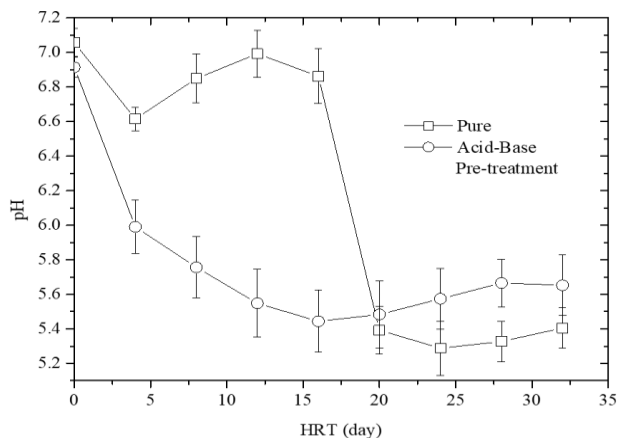


Figure 4. The effect of pH solution on AD processes in the biodigester under the mesophilic condition for 30 days

3.3 Sand characteristics for H₂S removal efficiency

The xFe₂O₃/cement-based sand specimen demonstrated a pH value of 6.54, a moisture content of 3.1% (w/w), and a porosity of 44.4%. Here, "w/w" denotes weight per weight. The biogas produced contained a minor concentration of H₂S, recorded at 11,140 parts per million (ppm). As depicted in Figure 5, the data illustrates the effectiveness of cement-based sand devoid of Fe₂O₃ in the removal of H₂S during a four-hour feeding period with varying flow rates. The cement-based sand achieved an H₂S removal efficiency exceeding 98% at a biogas flow rate of 59 mL per minute. Moreover, a negative correlation was observed between the flow rate and the amount of H₂S extracted from the biogas (Voegel et al., 2015; Lu et al., 2020), as shown in Figure 5. Nonetheless, even at the highest flow rate of 189 mL/min, an 80% reduction in H₂S was attained within a mere four-hour timeframe.

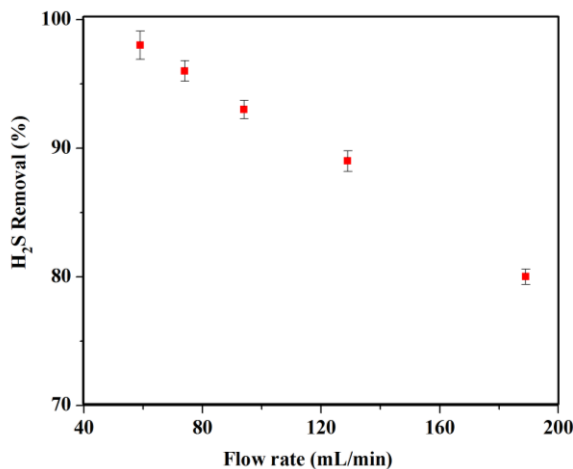


Figure 5. H₂S removal efficiency (%) of cement-based sand without Fe₂O₃ during 4 h of feeding time at a flow rate of 59–189 mL/min

As depicted in Figure 6, a flow rate of 59 mL/min successfully maintained a consistent 100% H₂S absorbance for the first 160 minutes of the experiment. However, this efficiency declined to 96% after 240 minutes. Conversely, H₂S absorbance in the cement-based sand, supplied at flow rates of 74, 94, 129, and 189 mL/min, gradually diminished to approximately 100% after 55, 50, 30, and 25 minutes, respectively, as illustrated in Figure 6. These results align closely with those reported in previous studies (Voegel et al., 2015; Pham et al., 2019), where an 80% reduction in H₂S removal efficiency was observed after a 6-minute water scrubbing process. This variation corroborates findings from earlier research (Lasocki et al., 2015), which demonstrated that the efficiency of H₂S removal using raw sand, acidic sand, typical sand, and podzol soil increased as gas flow rates decreased. Additionally, increasing the thickness of the bog iron ore layer enhanced H₂S removal efficiency, whereas biogas flow rates either decreased or remained stable (Carroll et al., 2016). The rise in biogas flow rate and the extended feeding duration led to the accumulation of H₂S and biogas, intended for household distribution. To achieve more sustainable biomethane production, it is crucial to optimize both H₂S and biogas levels.

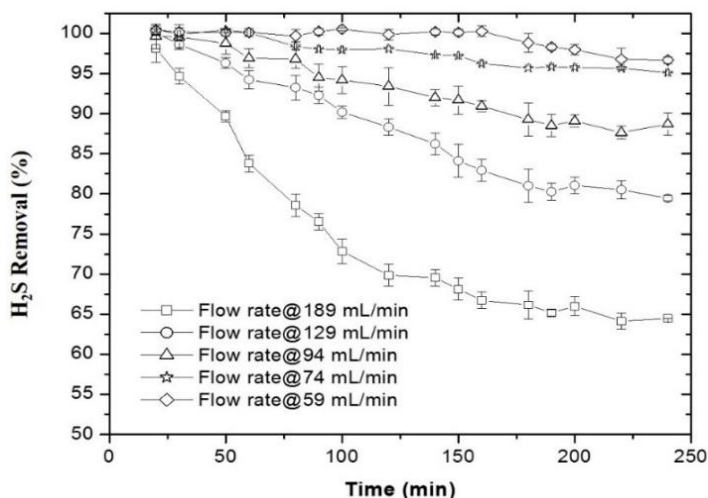


Figure 6. H₂S removal efficiency with increasing feeding time and biogas flow rates

3.5 Effects of Fe₂O₃ on H₂S removal efficiency

For this experiment, xFe₂O₃/cement-based sand was synthesized with varying Fe₂O₃ concentrations, ranging from 1% to 41.8% by weight. Figure 7 illustrates the effectiveness of H₂S removal using x Fe₂O₃/cement-based sand at two distinct flow rates: a low flow rate of 59 mL/min and a maximum flow rate of 189 mL/min. At a concentration of Fe₂O₃ below 2.5% in the cement-based sand, H₂S removal efficiency was less than 80% at the higher flow rate of 189 mL/min. Conversely, the absorbance and removal efficiency of H₂S ranged between 86% and 98% at the lower flow rate of 59 mL/min. Increasing the Fe₂O₃ concentration to 2.5% significantly enhanced H₂S removal at both flow rates (Figure 7). This phenomenon suggests an inverse relationship between the concentration of Fe₂O₃ in the cement-based sand and the removal efficiency of H₂S, irrespective of flow rate. H₂S removal efficiency was comparable between cement-based sand and Fe₂O₃/cement-based sand at a flow rate of 59 mL/min.

However, Fe_2O_3 /cement-based sand demonstrated the ability to maintain high H_2S removal efficiency across a broader range of flow rates (59 to 189 mL/min), whereas cement-based sand did not. The primary mechanism enabling Fe_2O_3 /cement-based materials to absorb H_2S is the reaction between ferric iron (Fe^{3+}) and the gas (Lasocki et al., 2015). In the Fe_2O_3 /cement-based sand absorber, the iron oxide (Fe_2O_3) reacts with H_2S as biogas flows through the column, forming iron sulfide (Fe_2S_3) and water (H_2O). The reduced H_2S removal efficiency in cement-based sand at higher flow rates may be attributed to the low moisture content, which is essential for maintaining Fe_2O_3 in its hydrated form (Carroll et al., 2016; Wu, 2020).

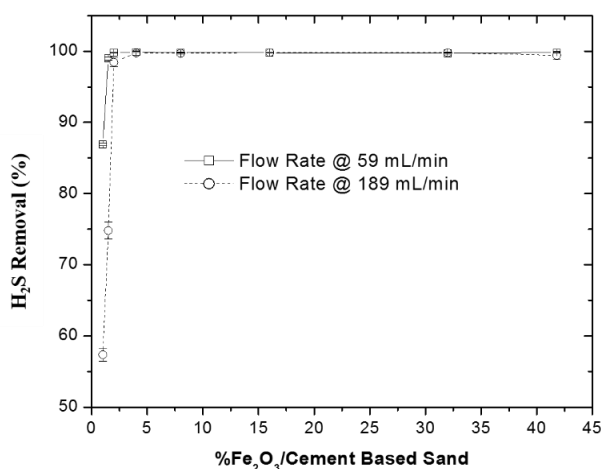


Figure 7. Tolerance of 4 wt.% Fe_2O_3 /cement-based sand against H_2S removal efficiency at the biogas rate of 59 and 189 mL/min

4. Conclusions

This research revealed that the acid-base pretreatment method was found to be most effective in breaking down lignin cross-links and making cellulose more accessible. After 16 days of exposure, it produced the most biogas, 84.84 mL/g, and the highest biomethane output, approximately 357.33 mL/g of VS. The initial pH of anaerobic digestion processes significantly impacts the activity of methanogens, a crucial factor in methane synthesis. The most effective absorber was cementitious sand with a 4% wt Fe_2O_3 content, effectively eliminating H_2S at gas flow rates ranging from 59 to 89 mL/min. The reusability of the Fe_2O_3 /cement-based sand could be significantly enhanced through washing and subsequent exposure to ambient oxygen.

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