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Evaluation of the use of a biofilter made with biodegradable material for biogas desulfurization

Christine Montemaggiore Becker*, Bruna Carolina Horn, Joice Mörs, Jeferson da Silva Couto and Odorico Konrad

Centro de Pesquisa em Energias e Tecnologias Sustentáveis, Universidade do Vale do Taquari, Av. Avelino Talini, 171, 95914-014, Lajeado, Rio Grande do Sul, Brazil. *Author for correspondence. E-mail: christine.becker@universo.univates.br

ABSTRACT. Biogas is a promising energy source with the potential to contribute to greenhouse gas emission reduction. In addition, it can be produced from waste biomass and has various applications. However, contaminants have to be removed before use. Hydrogen sulfide (H_2S) is perhaps the most critical because of its toxicity and corrosive properties. In this study, we evaluated a prototype of a biofilter for biogas desulfurization made of coconut chips as packing media and digester effluent as a nutrient source. The composition of inlet and outlet biogas was evaluated through gas chromatography in five tests. The maximum desulfurization efficiency observed was 75.80%, but with a considerable variation (mean of 43.08%). Microscopy analysis of the packing media before and after the experiments demonstrated the accumulation of substances and the presence of new elements. Nevertheless, more studies are necessary with longer duration and frequency for a better evaluation of the system stability and saturation.

Keywords: biogas; H_2S removal; desulfurization; biofilter; renewable energies.

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Introduction

There is a consensus in the scientific community that anthropic emissions affect climate change (Intergovernmental Panel on Climate Change [IPCC], 2021). At the same time, since the Paris Agreement in 2015, nations have committed to reducing avoidable greenhouse gas emissions and compensating them with carbon capture measures (United Nations Organization [UNO] & United Nations Framework Convention on Climate Change [UNFCC], 2015).

From all businesses, a study showed that the Energy sector has the most expressive contribution to GHG emissions (Cozzi, Gül, & IEA, 2021). In the same theme, one could point out the seventh Sustainable Development Goal of the 2030 Agenda: meet the growing demand for energy access for all communities without increasing air pollution (United Nations Organization [UNO], 2015).

In Brazil, the Energy sector is the second greatest emitter, corresponding to 450.58 $MtCO_2e$ or 31.04% in 2019[1]. However, sanitation activities also contribute to emissions, with solid waste management and wastewater treatment (Brasil, 2016b). The first is the second largest CH_4 emitter in 2018, with over 68 million tons[2].

In this sense, the scenario points to the need to increase the use of clean and renewable energies while seeking new alternatives for waste treatment. In general, bioenergy represents a suitable solution as it captures the atmosphere's carbon dioxide (Brasil, 2020; International Renewable Energy Agency [IRENA], 2020). Biogas, in particular, can be produced from waste biomass through anaerobic digestion (Brasil, 2016a; Brasil, 2016b; Kunz, Steinmetz, & Amaral, 2022). Hence, the technology is also a way of treating organic residues and wastewater close to the production unit, thus reducing the environmental footprint of transportation.

Another advantage is that biogas is an energy source with the potential to replace natural gas and other fossil fuels. Possible uses include heating, cooking, fueling, and electricity generation (Assunção, Mendes, Matos, & Borschiver, 2021; Irena, 2020; Karlsson et al., 2014). This is particularly relevant in developing

[1]Climate Watch Historical GHG Emissions. 2022. Washington, DC: World Resources Institute. Available online at: https://www.climatewatchdata.org/ghg-emissions

[2]Hannah Ritchie, Max Roser and Pablo Rosado (2020) - "CO_2 and Greenhouse Gas Emissions". Published online at OurWorldInData.org. Retrieved from: https://ourworldindata.org/grapher/methane-emissions-by-sector?country=~BRA.

countries since poorer populations tend to use energy sources that are more polluting and unsafe (Goldemberg & Lucon, 2012).

Biogas is a raw mixture of gas produced from the anaerobic digestion of organic matter. Its composition consists mainly of methane and carbon dioxide, but it can vary depending on the substrate digested (Agência Nacional Do Petróleo [ANP], 2015). One of the components of biogas is hydrogen sulfide (H_2S), which is highly toxic and corrosive (Esparza, Medina, Utrillas, Garcia, & Perello, 2019).

Therefore, for biogas to be used as an energy source, it is necessary to remove the H_2S, a process also called *desulfurization*. One of the technologies for that purpose is the biofilter or biotrickling filter, a fixed-bed reactor with adhered biomass (biofilm). Inside the biofilm, the sorption and biodegradation of the pollutant occur.

Reactors are filled with a packing media where biofilm will be attached (Kunz, Steinmetz, & Amaral, 2022). Examples of materials used as packing media are polymeric rings (Wu, Jiang, Jin, Yang, & Zhang, 2020), polymeric foam (Zeng et al., 2018b), and alternative materials, such as coconut chips or husk (Chaiprapat, Charnnok, Kantachote, & Sung, 2015; Tanikawa, Fujise, Kondo, Fujihira, & Seo, 2018).

Among the microorganisms capable of oxidizing H_2S, chemoheterotrophic bacteria *Thiobacillus* tend to require fewer nutrients (Abiogás, 2021). In general, sulfide-oxidizing bacteria (or SOBs) are part of the soil microbiota and produce sulfate from hydrogen sulfide. Sulfate can be absorbed later by plants, fungi, and other microorganisms (Madigan, Martinko, & Bender, 2016; Rocha, 2020).

Previous studies of this same research group revealed that, according to a literature review, biological methods tend to have relatively high and stable desulfurization efficiencies (Becker, Mader, Junges, & Konrad, 2022). Additionally, bio-desulfurization benefits are well known by Brazilian experts, according to whom these technologies have steady performance in several criteria (Becker, Horn, Oliveira, & Konrad, 2023, data not published).

Therefore, this study investigated the desulfurization performance of a pilot-scale biofilter. The prototype was built with biodegradable and readily available material, with coconut chips as packing media and digestate (digestion slurry) as a nutrient solution. Hence, the environmental impact of the end-of-life disposal of the materials decreases.

Material and methods

Tests were performed on a pilot scale at the Research Center on Sustainable Energies and Technologies (CPETS, in the original acronym) from the University of Taquari Valley, RS, Brazil. The biofilter was operated for 30 days exclusively with nutrient supply for microbial enrichment and adaptation of the biota. Subsequently, a series of experiments were conducted with biogas from an anaerobic digester for 15 days.

Experimental setup

The biofilter design had the premise of using biodegradable material and elements that could be incorporated into a composting plant. The setup consists of an acrylic cylinder of 0.1 m external diameter and 1 m of length, sealed with polymeric connections and silicon hoses for gas and liquid flows. Raw biogas was pumped from the bottom of the column and hence had an upward flow. Treated biogas (outlet gas) was collected at the top of the biofilter (Figure 1).

The biogas used in the experiments came from a domestic anaerobic digester, with a capacity of processing 1,200 L substrate and coupled with a 700 L gasometer. Digester was fed with malt bagasse and organic waste. Pumps of 6-12 V maintained the biogas flow, resulting in a flux of 0.23-0.25 $m^3\ h^{-1}$.

In a counter-current mode, a liquid flow of nutrients was sprayed daily at the top of the biofilter to keep the humidity and supply nutrients to the biofilm. Digestate, the effluent from anaerobic digestion, was used as a nutrient solution similar to other experiments (Chaiprapat et al., 2015; Dupnock & Deshusses, 2020; Zeng et al., 2019; Zhang et al., 2020).

Since the tests had a 1h duration, the nutrient solution was fed manually at the top of the packing media. A volume of 0.25 L per day was used both in the adaptation phase and immediately before each test.

As previously stated, an adaptation for this biofilter was conducted in the first 30 days. This was necessary to adjust the methodology, set the operational parameters, and grow and adapt the biofilm. The same solution used for nutrient supply was also the source of microbial enrichment, as reported by Zeng et al. (2019).

Coconut chips were the packing material of the biofilter. Chips are typically commercialized for gardening and are cut into pieces of approximately 1 cm (Figure 2). According to the manufacturer, the chips were

washed and had a pH of 5.5-6.5. The porosity was experimentally estimated according to Equation (1) (Metcalf, Eddy, Hespanhol, & Mierzwa, 2016), by weighing samples of the packing media before and after the adaptation phase.

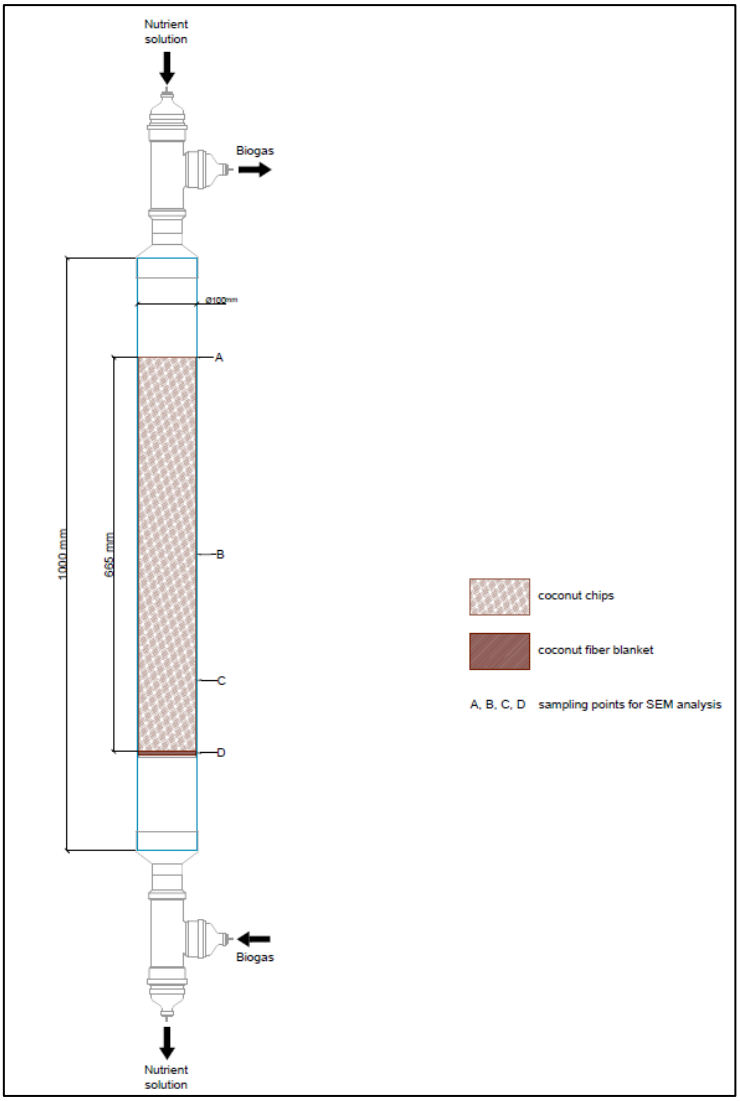

Figure 1. Experimental setup of the biofilter tested.

Figure 2. Coconut chips used as packing media before inoculation.

$$\propto_{chips} = \frac{V_h}{V_b} \times 100 = \frac{\frac{(w_w - w_d)}{\rho_{H2O}}}{V_b} \times 100 \quad (1)$$

Where:

α_{chips} = experimental porosity of the chips, %;

V_h = total hollow volume, cm^3;

V_b = bed volume, cm^3;

w_w = wet weight of the chips after the adaptation phase, g;

w_d = dry weight, g;

ρ_{H2O} = specific weight of water, g cm^{-3}.

As for the amount of packing media, the literature suggests a proportion of the packing media height/total biofilter height ratio of 1/3 up to 1 (Araújo, 2013; Zeng et al., 2019). The total bed height was 66.5 cm, resulting in a 2/3 ratio and a working volume of approximately 5 L.

Analytical methods

At each test, raw and treated biogas samples were collected in plastic bags. Biogas composition was determined by gas chromatography. The chromatograph was model Clarus 580 GC (PerkinElmer), equipped with Flame Photometric Detector (FPD, 325ºC, hydrogen as combustion gas) for measuring H_2S and Thermal Conductivity Detector (TCD, 250ºC, argon as carrier gas) for reading the concentrations of CH_4, CO_2, H_2, O_2, and N_2. The column was packed (Hayesep and molecular sieve), and the oven temperature was 60ºC.

The H_2S removal efficiency was calculated by Equation (2) (Metcalf et al., 2016, p. 1729). This was the parameter used for evaluating the desulfurization performance of the biofilter.

$$RE = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

Where:

RE = removal efficiency, %;

C_0 = H_2S concentration in the inlet biogas, ppm;

C_e = H_2S concentration in the outlet biogas, ppm;

As a benchmark for reactor construction that indicates the contact time between the biogas and the biofilter, the empty bed residence time was calculated using Equation (3) (Chaiprapat et al., 2015).

$$EBRT = \frac{V_b}{Q} \quad (3)$$

Where:

EBRT = empty bed residence time (s);

Q = inlet biogas flow ($m^3.s^{-1}$).

After the first series of tests, the effluent of a real-scale anaerobic digester (11.48% total solids and 81% fixed solids) was sprinkled at the top of the bed. The objective was to check for changes in the biofilm, though there was no further testing for hydrogen sulfide removal.

Subsequently, packing media samples were taken according to the sample points shown in Figure 1. They were analyzed using a Scanning Electron Microscope (SEM) Carl Zeiss EVO-LS10, coupled to Energy Dispersive Spectroscopy (EDS) for elemental evaluation of the surfaces.

Both coconut chips and coconut husk from the bottom of the bed were analyzed before and after the biofilter operation. Coconut husk was a fiber blanket used at the bottom of the biofilter to prevent the nutrient flow from dragging pieces of the biofilter. Samples were dried in a stove at 60ºC for 24h.

The digestate used as a nutrient solution and microbial source was characterized by measuring the acidity using a pHmeter Digimed – DM 2P. In addition, the total, fixed, and volatile solids contents were determined according to the methodology described by Hasan, Feitosa, Silva, Marder and Konrad (2019), which is a gravimetric method guided by *Standard Methods, 2450 Solids - 2450G*.

Results and discussion

Packing material and nutrient solution characterization

For porosity calculation using Equation (1), the material was weighed on an analytical scale before and after adaptation (when it was submerged in the nutrient solution). Results suggested a porosity of approximately 40%.

The pH of the digestate was slightly alkaline, 7.85 ± 0.55. The solid content is listed in Table 1.

Table 1. Solids content in the digestate.

Parameter	Values (%)
Total solids	0.34 ± 0.001
Fixed solids	58.56 ± 1.11
Volatile solids	41.44 ± 1.11

As to operational parameters, a literature review suggested a considerable variability among the values for inlet flow and empty bed residence time used by different authors (Table 2).

Table 2. Operational parameters used in biofilters in other studies.

Biogas inlet flow ($m^3\ h^{-1}$)	Empty bed residence time (s)	Working volume (L)	Source
0.0003 to 0.0013	60 to 240	2.50	Wu, Jiang, Jin, Yang, & Zhang (2020)
0.0120 to 0.0600	105 to 2100	5.000	Zeng et al. (2018a)
0.0240 to 0.0600	340 to 1710	11.40	Zeng et al. (2019)
0.4800 to 1.7400	18 to 180	17.70	Zhang et al. (2020)
0.2300 to 0.2500	68 to 75	5.07	Present study

Therefore, the biogas flow used here is greater than that adopted in other studies, Table 2. This may result in higher H_2S inlet loading and potentially affect the biofilter's performance. The empty bed residence time, however, was in the same range as the other experiments.

Evaluation of biogas composition

The outlet biogas samples were collected at different operation times. The times, H_2S concentrations, and other parameters are presented in Table 3. Figure 3 shows the variation in the composition (CH_4, CO_2, O_2, and N_2) of the outlet biogas ("*treated biogas*") compared to inlet biogas ("*raw biogas*").

Only in the first test, the CH_4 concentration was greater in the outlet stream. This suggests a possible dilution of biogas with air from the interior of the biofilter, which is also supported by the greater outlet O_2 concentration in the same tests. Anyway, there were no significant changes in methane concentration after the biogas flowed through the biofilter.

The increase in the nitrogen and oxygen concentrations in the outlet biogas may also indicate air inflow during sample collection. The outlet CO_2 concentration decreased minimally in the first three tests. There was a slight increase in the last two, even though these changes were also not expressive.

Variations in H_2S concentration between the inlet and outlet biogas are illustrated in Figure 4, and data are listed in Table 3. In all tests, the outlet concentration was lower, markedly on the second test. This sample was collected after 10 minutes of operation and had high oxygen content (as shown in Figure 3). From the results obtained, one could infer that starting at the tenth minute of operation, the biogas flow occurred from preferential pathways inside the unsaturated biofilter's bed.

Table 3. Data and parameters used in the present study.

Date	Time elapsed until sampling, min.	Inlet [H_2S], ppm	Outlet [H_2S], ppm	H_2S removal efficiency, %
August 16th	27	980.75	689.41	29.71
August 22nd	10	1315.52	318.37	75.80
August 24th	20	703.61	519.95	26.10
August 29th	30	746.90	418.47	43.97
August 30th	40	1030.52	620.21	39.82

Chaiprapat et al. (2015) evaluated biogas desulfurization at a plant that processed the effluent from a latex factory (initial H_2S concentration of 5,522 ± 1,371 ppm). The authors also used a biofilter made of coconut chips as packing material and digestate as a nutrient solution, both in single and triple-stage configurations.

The liquid flow, however, was continuous and kept at a pH lower than 4.0. In addition, the bed was previously inoculated with wastewater microorganisms.

The authors observed higher desulfurization efficiency on the triple-stage biofilter (69.0 to 96.7%). An increase in empty bed residence time from 100 to 180 s positively affected the performance (Chaiprapat, Charnnok, Kantachote, & Sung, 2015). Possibly, higher EBRT, combined with acidic pH, and the continuous recirculation of the nutrient solution, favored the desulfurization process on the biofilters evaluated by these authors.

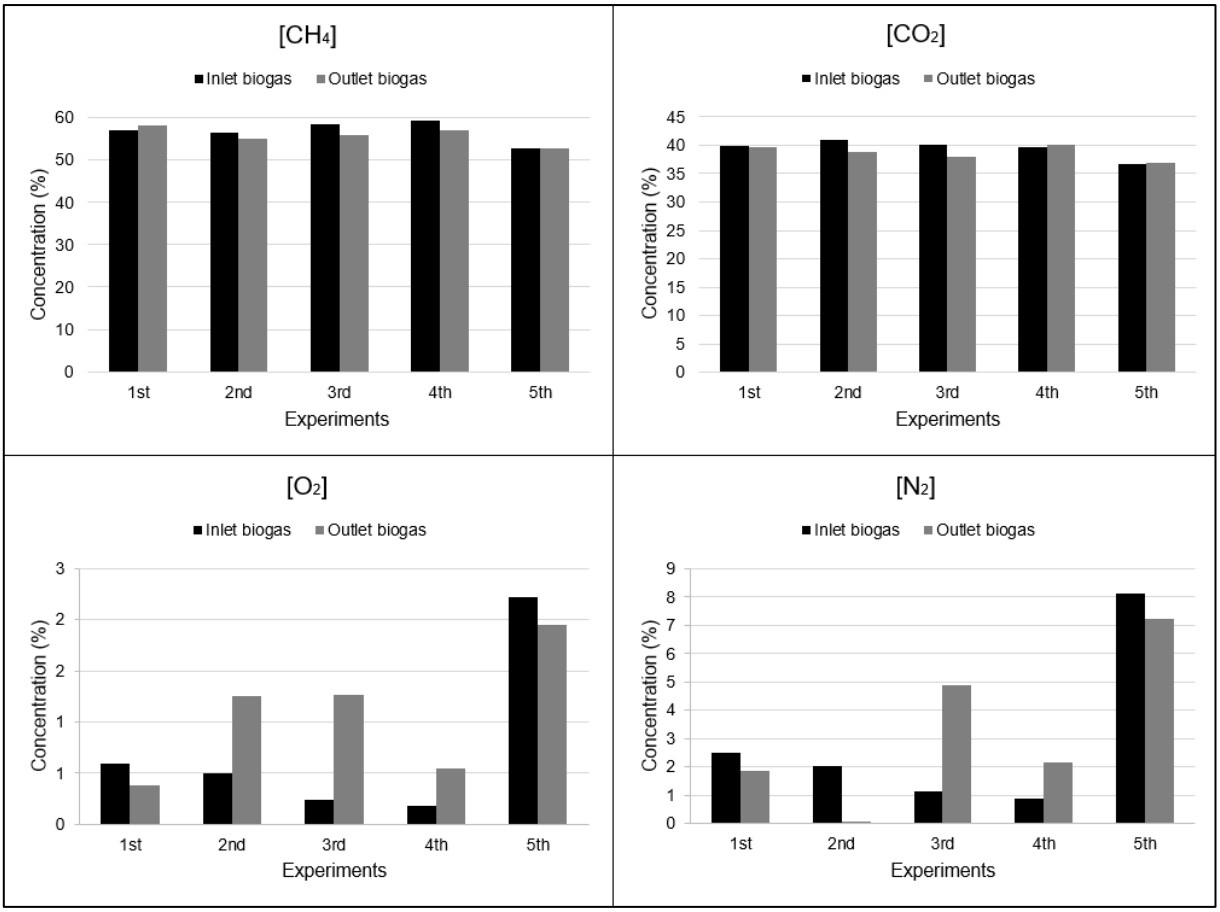

Figure 3. Comparison of inlet and outlet biogas composition.

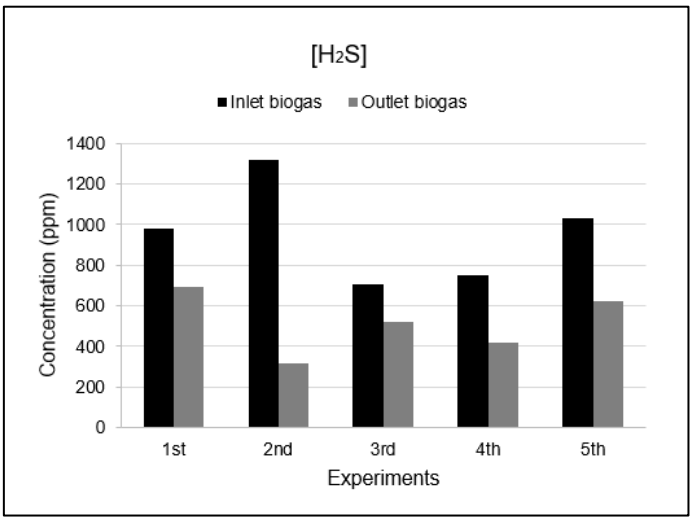

Figure 4. Comparison of inlet and outlet [H_2S].

On the other hand, some authors indicate that slightly alkaline conditions can increase the H_2S transfer rate from the gaseous to the liquid phase, thus increasing the performance (Wu et al., 2020). It has also been

reported that the use of digestate as a nutrient solution resulted in lower desulfurization efficiency compared to other nutrient solutions (Zeng et al., 2018a).

Hence, we suggest that further testing can better evaluate the technology. Additionally, it may be relevant to investigate the influence of parameters, such as pH, aeration, nutrient flow, empty bed residence time, and feeding mode (continuous or intermittent), possibly in tests with longer duration.

Microscopy of the packing media

Figure 5 illustrates the SEM of raw coconut chip and coconut husk samples before usage in the biofilter. Since these two samples had no moisture, they were not oven-dried before analysis. The EDS results indicated mainly the presence of minerals Na, Cl, and K in the chips. Coconut husk also had traces of Mg, Al, Ca, and approximately 21% of Si.

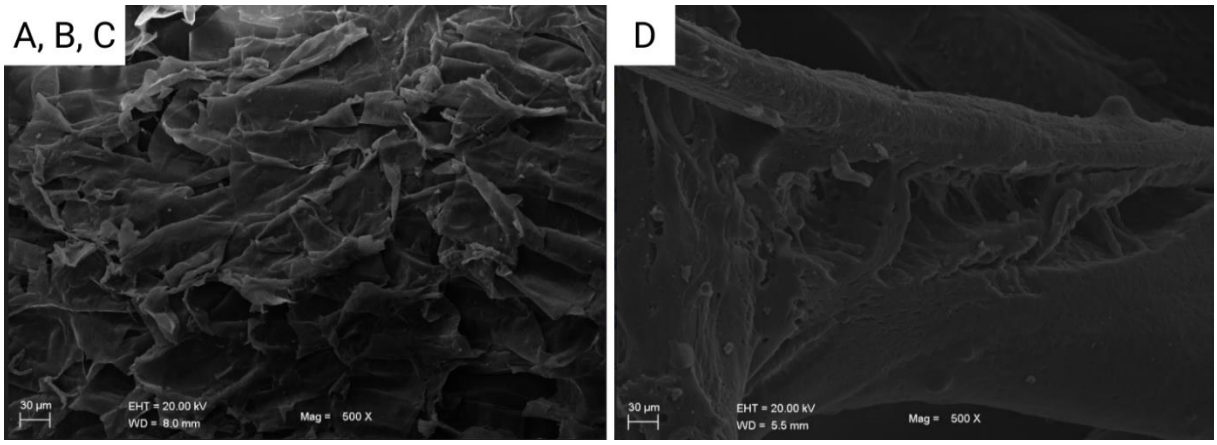

Figure 5. SEM images of coconut chips (A, B, C) and husk (D) before the tests.

After the operational phase, three additional samples of coconut husk and chips from three different heights were taken, and the results are shown in Figure 6. Noticeably, there was an accumulation of solids on the surfaces of all samples, and EDS results detected new elements (Table 4). Sulfur was detected in only one sample, the coconut husk from the bottom of the biofilter.

We emphasize, however, that the sample preparation for these analyses followed a different procedure than reported by other authors (Sahota et al., 2018; Zhang et al., 2022). During the drying period in the oven, it is safe to suppose that certain compounds were volatilized and hence were not quantified by EDS.

From the results in Table 4, most of the elements, like calcium, magnesium, and outstandingly phosphorus is inferred to come from the digestate of the real-scale digester, which was applied after the series of tests. The same applies to the silicon content since this second effluent applied to the bed had high fixed solids content.

As for the only sample that had sulfur content from the biofilter bottom, this was probably due to the nutrient solution flow. As it percolated the bed, the digestate may have dissolved and dragged the sulfur deposits to the bottom. Thus, sulfur would not be detected on the surface of the chips. Importantly, SEM analyzes samples with a surface area smaller than 1 cm^2. In this way, even with sample collection at different points, the sampling possibly did not include all the sulfur sludge deposits.

Table 4. EDS results for coconut chips and husk after biofilter operation.

Element	Amount detected (% weight)			
	Point A	Point B	Point C	Point D
Na - Sodium	4.01	-	5.70	4.55
Mg - Magnesium	2.06	4.12	5.20	6.10
Al - Aluminum	13.86	14.54	4.55	11.63
Si - Silicon	9.45	16.06	14.58	9.84
P - Phosphorus	5.55	2.05	-	3.26
K - Potassium	0.96	2.40	5.57	3.50
Ca - Calcium	22.14	31.79	36.56	4.44
Fe - Iron	40.83	18.10	24.96	29.11
Zn - Zinc	1.14	6.69	-	27.58
Cl - Chlorine	-	-	2.88	4.55
S - Sulfur	-	-	-	3.26

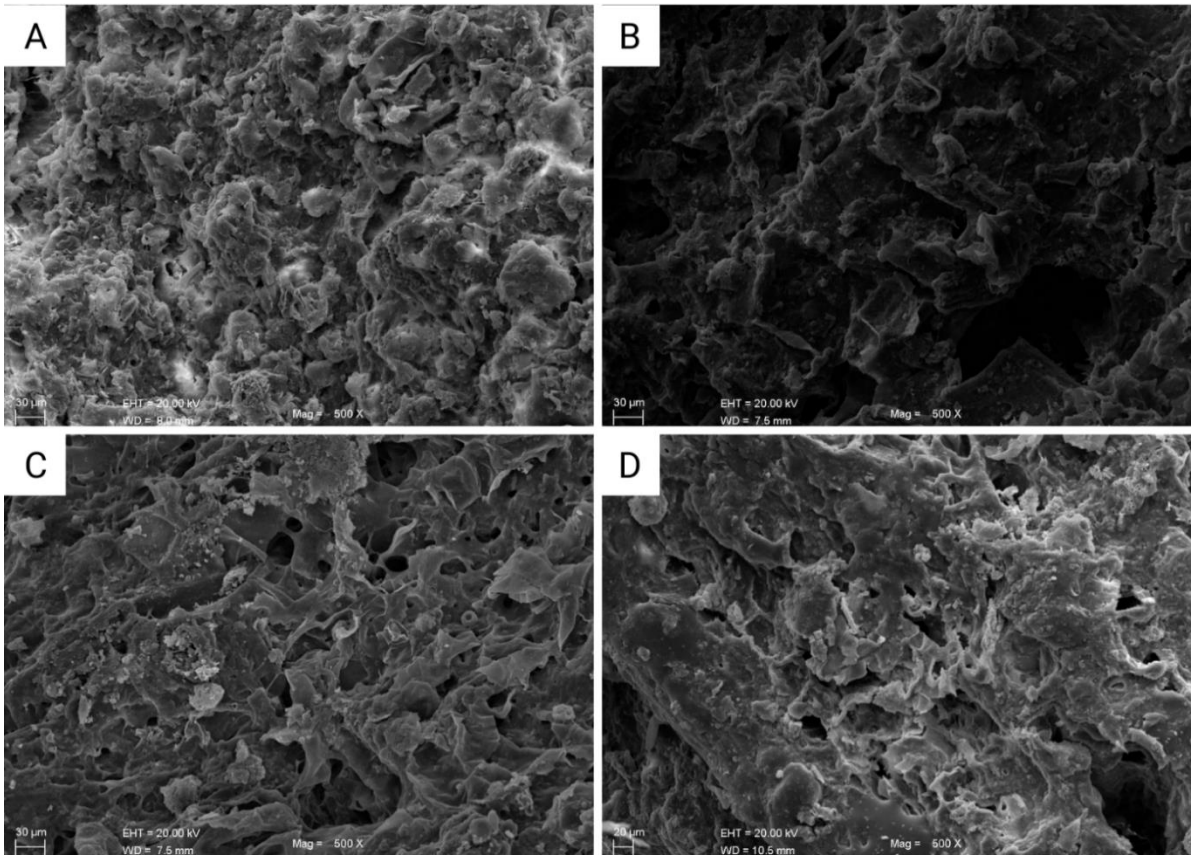

Figure 6. SEM images of coconut chips (A, B, C) and husk (D) before the tests, magnified 500x.

Conclusion

The present study reports a series of tests on biogas from anaerobic digestion, which evaluated the desulfurization efficiency of a biofilter. The system can reduce the H_2S content from biogas, with efficiency ranging from 26.10 to 75.80%. The highest efficiency was found on the sample of the outlet gas collected after 10 min of operation. In addition, variation in the results suggests the formation of preferential pathways for biogas flow on the reactor's bed.

The methane and carbon dioxide concentrations from the outlet biogas had minor variations. The presence of oxygen and nitrogen in some of the samples indicates the presence of air inside the biofilter. The SEM-EDS analysis proved that the pores of the packing media were filled with solid deposits, and the content of minerals and metals increased. One of the samples confirmed the presence of sulfur from the gaseous stream.

We recommend expanding the scope of studies on biofiltration, evaluating different pHs and liquid circulation modes (continuous or intermittent fluxes). With desulfurization tests using longer operational times, the hypothesis of the preferential flow paths could be tested. The inoculation with specific strains of sulfur-oxidizing bacteria is a strategy adopted by other authors that can improve the results since it potentially diminishes the competition with other microorganisms in the biofilm.

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