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Methane degradation in biotrickling filters: Recyclable materials as a support medium

Degradación de metano en biofiltros percoladores: Materiales reciclables como medio de soporte

Santiago Gómez Herrera. [1]; Yuly Alexandra Córdoba Diaz. [2]; Diana Carolina Morales Pabón.[3]; Hugo Ferney Leonel.[4]

1 *Universidad de Nariño, Colombia, sgh525@hotmail.com. https://orcid.org/0000-0002-4310-8420*
2 *Universidad de Nariño, Colombia. alexandra1995cordoba@gmail.com. https://orcid.org/0009-0008-0247-1275*
3 *Universidad de Nariño, Colombia, dianamoralespabon@udenar.edu.co. https://orcid.org/0000-0003-4552-074X (correspondende)*
4 *Universidad de Nariño, Colombia, huferleo@udenar.edu.co. https://orcid.org/0000-0002-6455-5182*

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ABSTRACT

Biofiltration is an alternative method for reducing methane, a greenhouse gas with public health risks and climate impacts. However, its feasibility is often limited by the high costs of organic beds and inadequate surface area. This study evaluated the removal efficiency and specific methane removal capacity of biotrickling filters (BTFs). In the air quality laboratory of the Universidad de Nariño, methane was diluted to a concentration of 4% in two BTFs with recycled material (polyethylene terephthalate - BTF1 and expanded polystyrene - BTF2) and inoculated with a methanotrophic microbial consortium. The reactors were operated in parallel for 24 hours per day with countercurrent flow under controlled conditions (25°C, 10 psi, neutral pH), TRLV of 31 min, and a flow rate of 6 L h-1 with a concentration of ~2000 ppmv. BTF1 achieved a maximum removal efficiency (RE) of 75%, higher than BTF2's 60%, likely due to the greater external specific area of PET. BTF1 and BTF2 showed a positive influence of temperature and humidity on RE, while pH had an opposite effect. However, BTF2 exhibited a higher specific removal capacity (SRC) due to its superior surface properties, though its performance was limited by filter bed compactation. In conclusion, BTFs using these two materials as support media demonstrate biological efficiency in methane removal, highlighting their potential for treating methane emissions from the anaerobic decomposition of organic matter in agricultural activities.

Keywords: air pollution; biofiltration; filter bed; recycled packaging; removal efficiency; specific removal capacity

RESUMEN

La biofiltración es un método alternativo para reducir el metano, un gas de efecto invernadero con riesgos para la salud pública e impactos climáticos. Su viabilidad se ve limitada por los elevados costos del uso de los lechos orgánicos y un área superficial inadecuada. Este estudio evaluó la eficiencia de remoción (ER) y la capacidad de eliminación específica de metano (CER) a través de biofiltros percoladores (BTFs). En el laboratorio de calidad de aire de la Universidad de Nariño, se diluyó el metano a una concentración del 4%, en dos BTFs con material reciclado (tereftalato de polietileno - BTF1 y poliestireno expandido - BTF2), inoculados con un consorcio microbiano metanotrófico. Los reactores operaron en paralelo durante 24 h.d-1, con flujo contracorriente y bajo condiciones controladas (25°C, 10 psi, pH neutro), TRLV de 31 min y un caudal de 6 L.h-1 con concentración de entrada de ~2000 ppmv. El BTF1 alcanzó una ER máxima del 75%, superior al 60% del BTF2, atribuida posiblemente a la mayor área superficial específica externa del PET. En ambos BTF, la temperatura y humedad influyeron positivamente en la ER, mientras el pH tuvo un efecto contrario. Sin embargo, el BTF2 mostró mayor CER, atribuida a sus propiedades superficiales superiores, aunque su desempeño se vio limitado por compactación del lecho. En conclusión, los BTF que emplean estos materiales como medio de soporte muestran una alta eficiencia biológica en la degradación del metano, lo que resalta su potencial para el tratamiento de emisiones generadas por la descomposición anaeróbica de materia orgánica en actividades del sector agropecuario.

Palabras clave: biofiltración; capacidad de eliminación específica; contaminación atmosférica; eficiencia de remoción; empaque reciclado; lecho filtrante.

INTRODUCTION

Methane (CH_4) is one of the most abundant hydrocarbons in nature and the main component of natural gas. It is currently recognized as the second most important anthropogenic Greenhouse Gas (GHG) in the atmosphere (Lancon & Hascakir, 2018; Soeder, 2021). DeFrabrizio *et al.* (2021) state that methane emissions have increased by 25% over the past 20 years. Its global warming potential is 28 times higher than that of CO_2, with a half-life of 12 years in the atmosphere, accounting for approximately 20% of global emissions (Sanucci, 2021).

Different alternatives for controlling methane emissions have been explored, including incineration or valorization processes, which are the most commonly used for high concentrations (>30%) (Gómez-Borraz *et al.*, 2017; Borchardt *et al.*, 2018). The operation of organic and inorganic biofilters has also been analyzed as a feasible and stable method for treating diffuse methane emissions (Gómez-Borraz *et al.*, 2017). Other mitigation practices include livestock feed additives, new rice cultivation techniques, advanced approaches to oil and gas leak detection, and modern water and waste facilities, which are reported by DeFrabrizio *et al.* (2021). However, they also note that many of these solutions have not been widely adopted due to high costs or a lack of awareness about available technologies. Additionally, Reza Bacelis *et al.* (2009) indicated that methane biological oxidation is a process in which natural systems and soils, particularly those rich in organic matter, break down methane.

In this context, bio-percolation is gaining relevance as an alternative for low-concentration emissions, as it is one of the most profitable configurations due to its robustness (Zimmermann *et al.*, 2021). This biotechnology relies on microorganisms, particularly methanotrophs, to oxidize CH_4 to generate less harmful products such as water, carbon dioxide (CO_2), biomass, and salts (Mayer *et al.*, 2019). However, the successful operation of a biofilter depends largely on the type of media support and its physicochemical characteristics, as these factors are fundamental to the optimal development of the biofilm. Generally, these beds are composed of organic materials such as soil or compost, which serve as nutrient sources (Khabiri *et al.*, 2022). Nevertheless, these materials have been shown to have a short lifespan (<6 months) and are prone to long-term issues such as clogging and pressure drops (Cassarini *et al*., 2019a; Cassarini *et al*., 2019b). In this regard, Sáez-Orviz *et al*. (2024) confirmed that reused and recycled plastics from the integral water cycle could effectively serve as packing material in bio-trickling filters (BTFs), reducing odors by 90%.

On the other hand, beds composed of inorganic materials (e.g., rocks, ceramics, glass) offer several advantages, as their physical properties can be easily adjusted. They exhibit more stable long-term behavior and provide good mechanical properties (Cáceres *et al.,* 2017). In recent years, one of the main challenges in their implementation has been their relatively high costs compared to organic packaging, as well as their unsuitable surface for biomass formation (Gómez-Cuervo *et al*., 2017). To assess the performance of these systems, key monitored parameters included temperature, relative humidity, and the pH of the leachate from each BTF. Methane concentration was measured daily using samplers positioned at the system's inlet and outlet. In this context, of the current trend favoring integral waste management, it is important to note that there are no reports on the use of recyclable materials as support media, which could improve the operational profitability of these systems. Therefore, this study evaluated the effectiveness of bio-trickling filters (BTFs) in removing methane from a gaseous stream. It utilized a support bed made from inoculated recycled materials, specifically expanded polystyrene packaging and polyethylene terephthalate bottles.

MATERIAL AND METHODS

Biofilter design and operation

Based on scientific literature and technical specifications (Wu *et al*., 2018), two identical BTFs were designed using transparent acrylic tubes with a volume of 6.2 L, a useful height of 0.6 m, and an internal diameter of 0.115 m. A sampling point was located at a height of 0.3 m. Each BTF was packed with approximately 30 cm of selected recycled materials. BTF1 contained polyethylene terephthalate (PET) bottles, which were arranged in 2 cm-thick strips, forming rosettes, each measuring 10 cm in length. BTF2 expanded polystyrene was used, sourced from packaging cubes with an edge length of 3 cm. Before use, all materials were washed with distilled water to remove any surface impurities.

The treated gas stream consisted of a mixture of air from a compressor and pure methane from Cryogas-Air products (Colombia), regulated by Cole Palmer Instruments, USA, rotameter-type flow controllers. Additionally, the gas phase flowed in an upward direction, while the percolating liquid moved counter-currently through a distributor plate, ensuring uniform wetting of the support material (Su *et al*., 2023). An empty bed contact time (EBCT) of 31 min and an inlet flow rate of 6 L h-1 were used, with methane concentration varying approximately between 4% vv-1 and 4.5% vv-1 of CH4, which corresponds to pollutant loads (PL) of 1.65 to 2.20 gCH4 m-3 h-1.

The research was conducted at the Air Quality Laboratory of Universidad de Nariño, where a closed, greenhouse-type compartment was built to isolate the assembly to automatically maintain a temperature of 25°C, with a K-type thermocouple and a heater. In addition to controlling the gas pressure of 10 PSI, the system was equipped with manometer-type pressure regulators (Figure 1).

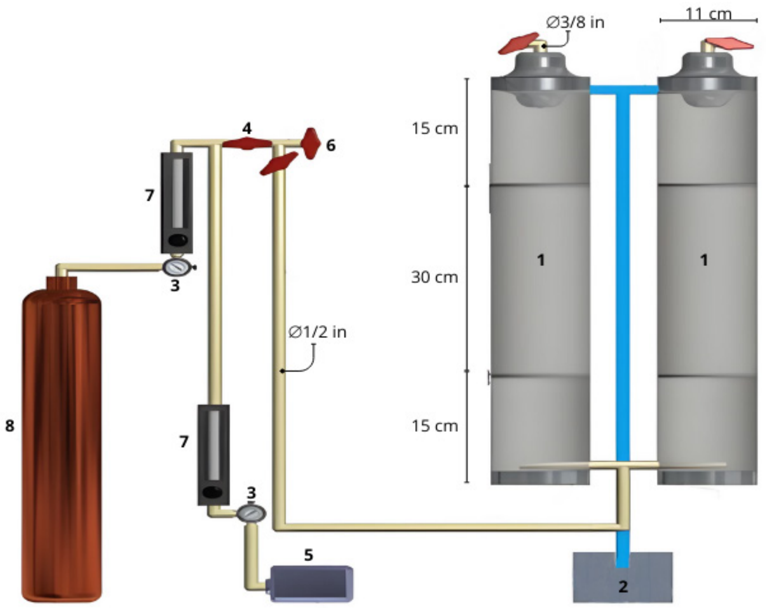

Figure 1. *Schematic representation of the experimental set-up.*

*(1) Biotrickling filter, (2) recirculation pump, (3) manometer, (4) mixing chamber, (5) air compressor, (6) inlet sampler, (7) flowmeter, (8) methane.

The monitored parameters included temperature, measured with a Peakmeter K-type thermocouple (China); relative humidity determined using the High-Tech Instruments (China); and the pH of the leachate from each BTF with the Pinpoint (USA). All equipment had factory calibration certification. Methane concentration was measured daily using a Vetus semi-conductor portable measuring instrument (The Netherlands), with samplers installed at the inlet and outlet of the system.

Once the steady state of the removal efficiency (RE)—defined as a variation of 10% to 5% over consecutive days—was reached, as stated by Gómez-Borraz *et al.* (2017), Jawad *et al.* (2021), and Khabiri *et al.* (2022), 56-point samples were taken every 12 h. These samples allowed for the determination of elimination capacity (EC) in gCH4 m-3 h-1 and specific elimination capacity (SEC) in gCH4 m-2 h-1 at pollutant loads (PL) around 4% CH4 concentration. These parameters were calculated using the following equations:

$$PL = (CCH_4)_{Inlet} x \frac{Q}{V}$$

$$RE = \frac{(CCH_4)_{Inlet} - (CCH_4)_{outlet}}{(CCH_4)_{Outlet}} x\ 100$$

$$EC = (CCH_4)_{Inlet} - (CCH_4)_{outlet} \frac{Q}{V}$$

$$SEC = \frac{EC}{As}$$

Where CCH4 is the methane concentration in g.m-3; Q is the volumetric flow rate of the gaseous stream m3.h-1; V is the volume occupied by the filter media in m3, and As is the specific area of each material in m2.

Microorganisms and culture conditions

During the study, 1.5 L of mineral salt medium was recirculated in each BTF as a nutrient source for the biomass, using a Sunsun Group-type submergible water pump (China). This solution was prepared in distilled water, following a method similar to that described by Lebrero *et al.* (2019), with a final pH of 7 and renewed weekly (Table 1).

Table 1. *Mineral salt medium*

Reagent	Concentration (g.L^{-1})
$NaNO_3$	2.0
$MgSO_4.7H_2O$	0.2
$FeSO_4.7H_2O$	1×10^{-3}
Na_2HPO_4	0.2
$NaH_2PO_4.H_2O$	0.09
$CoSO_4.5H_2o$	5×10^{-6}
H_3BO_3	1×10^{-5}
$MnSO_4.5H_2O$	1×10^{-5}
$ZnSO_4.7H_2o$	$7x10^{-5}$
MoO_3	$1x10^{-5}$
KCl	0.04
$CaCl_2$	$1.5x10^{-3}$

Both support materials were inoculated with 240 g of Evogen P.C.H (Genesis Biosciences, USA), a mixture of various strains of aromatic and aliphatic hydrocarbon-degrading bacteria (a consortium of *Pseudomonas* bacteria species). In addition, the mineral medium was continuously recirculated at a flow rate of 140 mL min-1 for two weeks, along with the addition of methanol as the sole carbon source, and an air flow of 0.006 m3 h-1 to promote microbial growth (Gómez-Borraz *et al.*, 2017; Venturini *et al.*, 2022).

Subsequently, during the acclimatization process, the system was fed with a 2% methane stream, and methanol was excluded from the mineral medium. This process lasted two weeks to allow biofilm growth on the packing material. During this initial period, at the end of each week, biomass was recovered from the mineral medium by centrifugation (4000 rpm for 10 min), then resuspended in fresh medium and reintroduced into the system. Once the system stabilized, the synthetic nutrient solution was supplied twice daily for 20 minutes at a flow rate of 8.5 L h-1 to maintain adequate moisture in the filter bed and to provide essential nutrients for microbial growth.

Statistical analysis

The experimental design was entirely randomized fixed-effects model. sampling was probabilistic, based on the determination of sample size (power >95%), ensuring the randomness of the process. Normality and homoscedasticity tests were performed, which led to the selection of the Kruskal-Wallis test (α=0.05) to estimate differences between treatments.

To determine the influence of confounding factors on the removal efficiency of each BTF, Principal Component Analysis (PCA) (α=0.05) was conducted using a correlation matrix, along with a probabilistic multiple regression model adjusted by ordinary least squares.

RESULTS AND DISCUSSION

During the start-up period in both BTF1 and BTF2, lower bio-oxidation was evident, which may indicate that a greater amount of the carbon source was used for biomass growth than for oxidizing CH_4 to CO_2, similar to findings of Gómez-Cuervo *et al.* (2017). The change from the acclimatization phase to the operation phase likely created favorable conditions for the development of methanotrophs, which required a higher methane concentration to promote biomass. This, in turn, led to a more stable performance with lower effluent methane concentrations (Ferdowsi *et al.*, 2022).

Figure 2 shows the EC of each BTF, and Figure 3 shows the RE of each BTF.

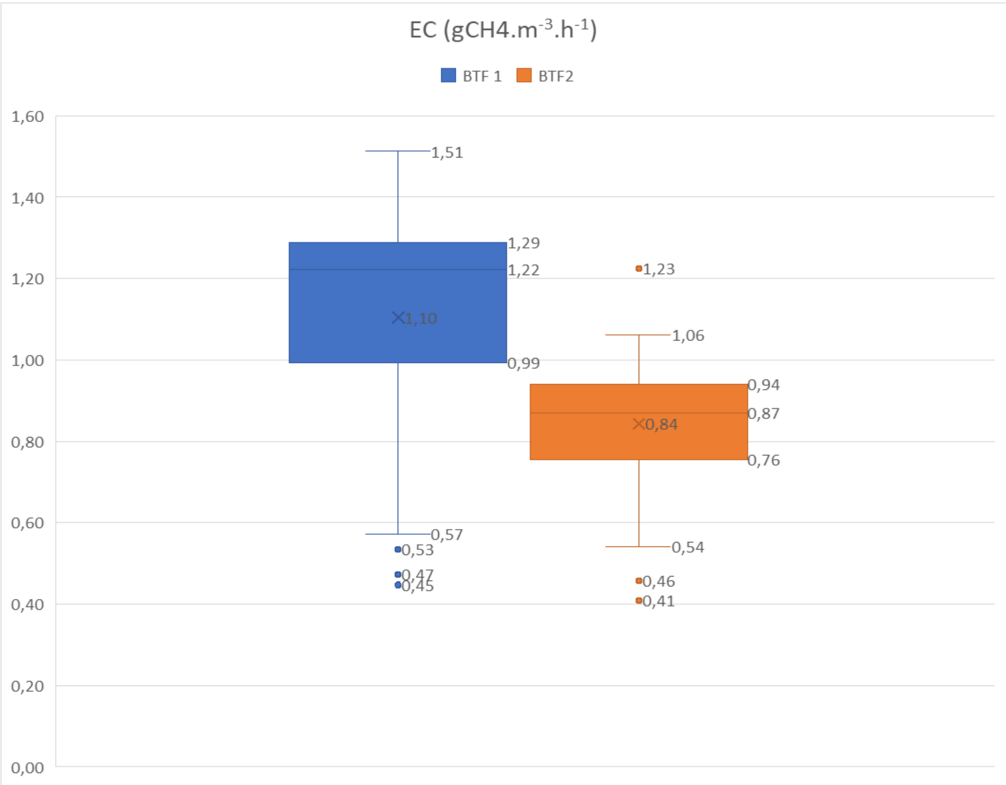

Figure 2. *Elimination Capacity (g CH4 m-3.h-1) by BTF1 and BTF2*

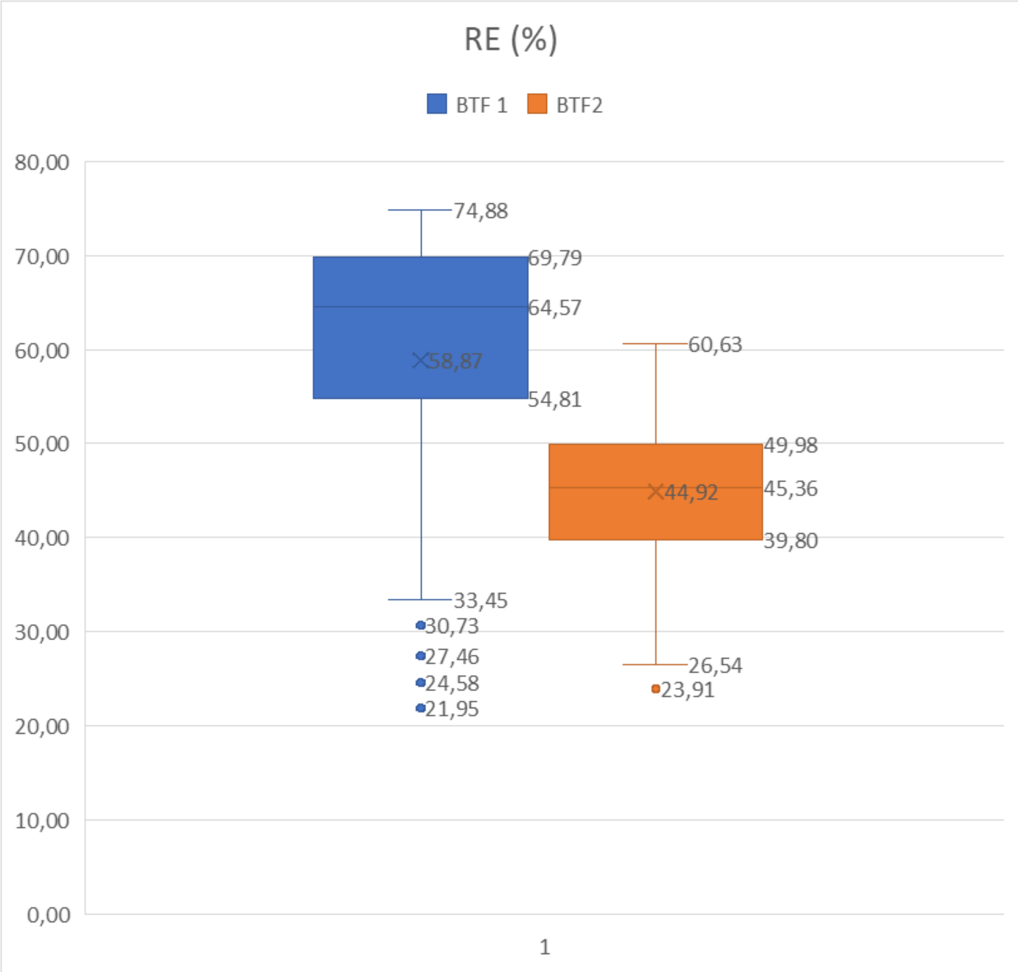

Figure 3. *RE % by BTF1 and BTF2*

As mentioned above, the biofilter packed with recycled plastic rosettes exhibit better performance, allowing for greater gas flow stability and a larger biofilm surface area. This resulted in an increased transfer of contaminants from the gas phase to the biofilter, ultimately leading to higher RE. (Figure 3).

Compared to other studies using similar materials, the maximum REs were high, which may be attributed to the EBCT enhancing gaseous mass transfer, thereby facilitating CH_4 solubilization in the aqueous phase and increasing its availability to methanotrophs (Gómez-Cuervo *et al.*, 2017). Furthermore, Merouani *et al.* (2022) found a clear tendency for better results at low inlet flows, as inhibition occurs more easily at higher CH_4 concentrations than at lower ones. In other words, high flow rates reduce contact time between the pollutant and the microbial population, accelerating the system's critical load (Liu *et al.*, 2020b; Khabiri *et al.*, 2022).

Concerning the External Specific Area (As), recycled PET was almost 50% larger than the expanded polystyrene cubes, with a difference of 22% in the EC. This suggests that, for the materials evaluated, As may not be the only determining factor in achieving high levels of removal. This aligns with the findings of Khabiri *et al.* (2020b), who state that for variations greater than 10%, there should be differences of at least 30% in removal capacity. On the contrary, L. Cai *et al.* (2019) and S. Cai *et al.* (2020) mention that the attachment of microorganisms to a solid packaging material depends on several surface properties. These may include surface roughness, hydrophobicity (contact angle), surface charge, or the production of extracellular polysaccharides (EPS) by microorganisms.

Now, for the SEC, BTF1 reached a maximum of 0.12 gCH4 m-2 h-1 and an average of 0.094 gCH4 m-2 h-1; while BTF2 had a maximum was 0.15 gCH4 m-2 h-1 with an average of 0.11 gCH4 m-2 h-1. The data collected reveal that higher inlet loads are related to higher EC values, with BTF2 achieving values up to 150% higher than BTF1 in the start-up period. Consequently, high EC values are associated with good surface properties, which favor the binding of active methanotrophs and the development of a biofilm (Khabiri *et al*., 2020a). The ECS represents the actual CH4 removal efficiency associated with a given external surface area of the support medium (Cáceres *et al*., 2017). Therefore, since this measure is not As-dependent, it allows comparison of the performance of the two beds based solely on their surface properties.

If the EC of BTF1 is higher than the EC of BTF2, it is mainly due to its higher As value. This suggests that if the As of BTF2 were increased (for example, by using less porous expanded polystyrene cubes), it could theoretically perform as well as BTF1, given its superior surface properties. Compared to other research, the SEC values in this study are higher than those presented by Cáceres *et al*. (2017), who obtained 0.019 gCH4 m-2 h-1 using 316 m-1 polyethylene rings, and higher than those reported by Liu *et al*., (2020a) who recorded a maximum value of 0.037 gCH4 m-2 h-1 using 600 m-1 polyurethane foam.

That said, it is important to mention that in the first 20 samplings, the EC of both BTFs remained similar. Biofilm development occurred first on BTF2, perhaps due to its roughness properties, which provide an increased surface area for biofilm attachment and adhesion, making detachment less likely (Carabelli *et al*., 2022). Despite this, material compaction processes influenced the reduction in cube size, leading to a decrease in the amount of microorganisms present. This aligns with previous findings showing that bacteria are more easily removed from young cultured biofilms (Carabelli *et al*., 2022; Sauer *et al*., 2022).

On the contrary, the plastic rosettes showed a slower start-up because their smooth surface did not allow complete attachment of the microorganisms (Gassman *et al.,* 2022). However, in the end, their larger surface area allowed sufficient biomass to be harbored to achieve considerable removal rates. These results are in agreement with the findings of (Cáceres *et al*., 2017), where the highest SEC values were recorded for the particles with the highest specific surface area.

Temperature

For BTF1, the average temperature was 28°C, while for BTF2, it was 26.5°C, with minimum temperatures of 26°C and maximum temperatures of 31°C in both biofilters. The temperature differences may be due to microbial activity, as all biooxidation reactions are exothermic, and the energy released by these reactions causes a positive temperature gradient in the packed bed (Lebrero *et al.,* 2021). Therefore, microbial intensity in the support medium is strongly dependent on the temperature of the BTF (Chaghouri, 2021).

On the other hand, Wu *et al*. (2018) state that temperature has two important effects on mass transfer: an increase in diffusion coefficient, which facilitates the transfer of the pollutant to the biofilm, and the Henry coefficient, which reduces the solubility of the compound. Therefore, the balance between these two effects can determine the optimal performance of a biofilter.

T, HR, pH, and RE were the follow-up variables for the PCA, which was conducted using a Pearson's test (*P*=0.05). About RE, the results confirm that the samples with the highest efficiencies occur at temperatures above 29°C, with high positive correlations found for the BTFs in the PCAs (0.93 BTF1 and 0.91 BTF2) as shown in Figures 4 and 5. Temperature was the most influential monitoring parameter. In BTF1, at high temperatures such as 30 and 31°C, removal efficiencies of 73.91, 74.19, and 74.88 % were observed. In BTF2, removal efficiencies of 60.63 and 60.44% were achieved at temperatures of 29 and 31°C.

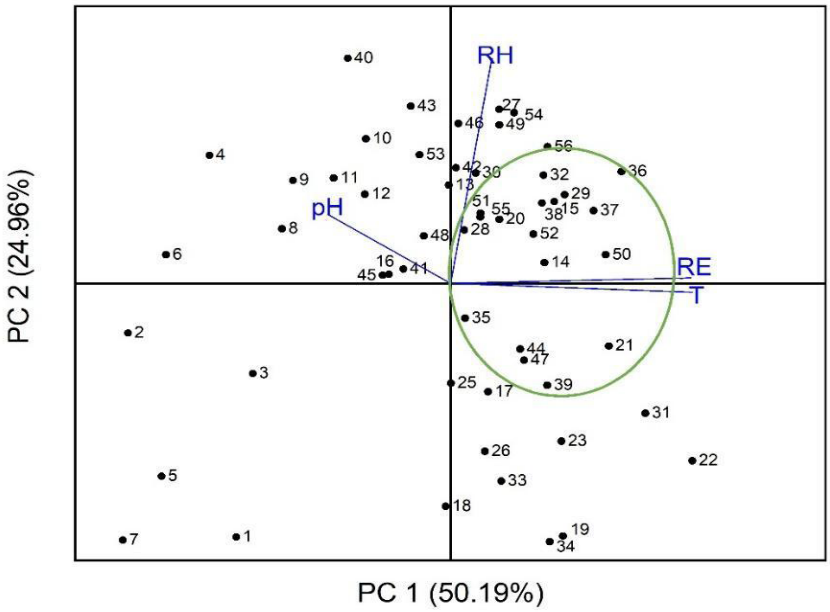

Figure 4. *Principal Component Analysis BTF1*

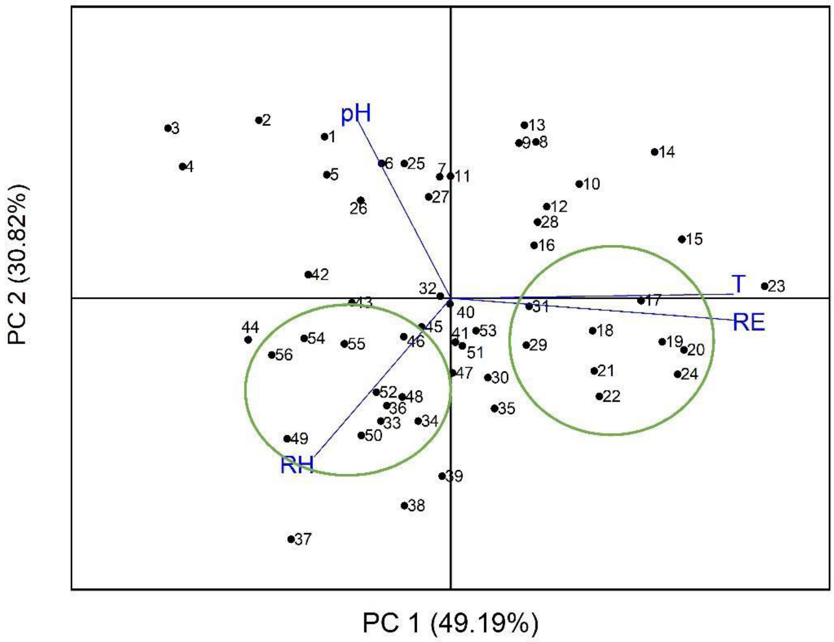

Figure 5. *Principal Component Analysis BTF2*

According to Figures 4 and 5, the PCA analysis of BTF1 (Figure 4) and BTF2 (Figure 5) indicates that the first principal component exhibits a substantial correlation with temperature (T), relative humidity (RH), and removal efficiency (RE) in both biofilters. However, RH has a more significant negative effect on BTF2 (Figure 5), suggesting that material properties, such as expanded polystyrene, influence biofilter efficacy.

In general, the systems operated under adequate temperature conditions that supported high microbial growth and efficiency, which is consistent with the findings of Jugnia *et al.* (2012), who conclude that optimal temperatures for high RE are between 25 and 31°C. Likewise, Gómez-Borraz *et al.* (2017) found that in biofilters packed with polyurethane sponge and Raschig rings, the highest methane consumption rates were obtained for tests conducted at temperatures of 25 and 30 °C.

pH

The pH showed little variation, ranging from 6.2 and 7.02, which is considered optimal for microbial growth. According to Pratt & Tate, (2018) and Nisbet *et al.* (2020), the pH should be very close to neutral. Vikrant *et al.* (2018) suggest that these pH values may be explained by the buffering effect of the nutrient mineral medium, because it was renewed weekly, it prevented rapid pH decay.

Filter bed pH has been considered as a parameter of low importance because CH4 biodegradation does not generate intermediate or end products capable of significantly influencing pH (Thomasen *et al.*, 2019; Pecorini *et al.*, 2020). However, abrupt changes in this variable may reflect lower methane removal rates, as shown by the negative correlations for both BTFs (-0.5 and -0.3, respectively). Gómez-Cuervo *et al.* (2017) found that acidification in a biofilter, associated with the use of ammonium as a nitrogen source, causes pH instabilities (pH <5) and reductions in RE.

Relative Humidity

The minimum relative humidity in BTF1 was 68.5% with a maximum of 76.5% and an average of 73.6%. For BTF2, it was 74.8% with a maximum of 97.1% and an average of 84.79%. The PCAs indicate a difference in how relative humidity influenced each BTF. While BTF1 exhibited a positive correlation with RE (0.95), due to its stability throughout the study, BTF2 showed a negative correlation due to the difficulties of the support material (-0.73).

According to Gómez-Cuervo *et al.* (2017), a lack or excess of water can cause significant reductions in removal values, and it is estimated that 75% of biofilter failures result from one of these two factors. Domingues *et al.* (2021) state that the recorded humidity ranges are not ideal for optimal inorganic biofilter performance, which should be between 30% and 70%. Excess humidity can cause water blockage, leading to lower RE, with higher water accumulation negatively affecting CH4 mass transfer. This effect was observed by Gómez-Cuervo *et al.* (2017) in a biofilter packed with polyurethane foam.

For BTF2, the dripping from the irrigation system caused a constant silting of the bed, leading to grooving and a reduction in contact area, which resulted in a decrease in efficiency of up to 26% from sample 25. Inadequate relative humidity content favors high head losses, increased resistance to mass transfer, leading to the anaerobic zones and excessive leachate production. Additionally, it creates stressful conditions for the biomass and its metabolism (Khabiri *et al.*, 2020a;

Pecorini *et al*., 2020). In BTF1, this issue did not occur, as its bed is composed of resistant plastic material that is difficult to compact.

For the development of the model, four independent variables affecting RE (dependent variable) were identified: type of biofilter (BTF1 or BTF2), temperature, pH, and relative humidity. Since the type of biofilter is a qualitative variable, it was coded using Dummy variables, assigning 0 to BTF1 and 1 to BTF2 (Mayhew & Simonoff, 2015). According to ANOVA, with a P-value < 0.05 and R^2 = 78%, the independent variables were found to be significantly associated with RE. The model predicts that BTF2 is 11.52% less efficient than BTF1, while increases in temperature and relative humidity improve removal by 7.6% and 0.06%, respectively.

CONCLUSIONS

The selected recycled materials offer significant advantages over traditionally used media due to their suitable physical properties and ease of collection, which do not present major challenges. Furthermore, they do not generate significant costs that would hinder their adoption. The results indicate that recyclable materials can be a viable option for improving the efficiency of these systems. However, PET plastic rosettes appear to be more efficient than expanded polystyrene cubes due to their higher specific surface area, which promotes greater biofilm development. On the other hand, BTF2, due to its roughness, appears to have better surface properties, allowing for faster microorganism adhesion during the boot stage.

The strong positive correlations observed in the PCAs indicate that temperature is the most influential variable concerning methane biofiltration process, as its serve as a reliable indicator of microbial activity and its degradative action. Regarding relative humidity, the study found that the excess moisture negatively impacts methane removal, as it inhibits microbial metabolism and promotes silting in porous beds.

Throughout the study, pH remained neutral, confirming an optimal biofiltration process, as neutral pH conditions are favorable for methanotrophic biomass. However, when the pH fluctuated sharply, an inverse relationship with removal efficiency was observed.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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