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EXPERIMENTAL RESEARCH ON THE SELECTED BIOFILTRATION MATERIALS UNDER DYNAMIC CONDITIONS

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Abstract. Hydrogen sulfide (H_2S) is a hazardous chemical compound present in raw biogas and requires removal. Biofiltration offers an eco-friendly solution by utilizing sulfur oxidizing bacteria (SOBs) within a biofilter. This biofilter typically comprises packing material to house SOBs and facilitate desulfurization. To optimize removal efficiency (RE), the physicochemical properties of packing materials (organic/inorganic/synthetic) need evaluation. This study focused on the characteristics of sewage sludge and biochar samples produced via pyrolysis at temperatures of 400 °C, 500 °C, and 600 °C, along with cellular concrete (CLC) waste and polyurethane foam (PUF). Measurements included bulk density, pH, and electrical conductivity, with discussion on their impact on H_2S purification from biogas under dynamic conditions. Ultimately, PUF, CLC waste, biochar after 600 °C pyrolysis, and sewage sludge exhibited superior performance in terms of lowest bulk density, optimal pH, and highest electrical conductivity.

Keywords: H_2S removal, biofiltration, packing materials, physicochemical properties.

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1. Introduction

Hydrogen sulfide, certainly one of the most harmful substances found in raw biogas, can make various challenges for the environment and human health if not removed before the primary gas is utilized for electricity generation (Abd & Othman, 2022). Several techniques are utilized to purify H_2S from biogas (Gao et al., 2022). Among all, the most environmentally friendly and efficient method for desulfurizing hydrogen sulfide from biogas is the utilization of a pilot-scale biofilter (Khalil et al., 2019). A critical component of a typical biofilter, which significantly influences its removal efficiency (RE), stability, and ability to maintain specific environmental conditions for optimal operation, is the choice of packing materials (Juntranapaporn et al., 2019). In a typical biofiltration process, a bed of organic or inorganic porous materials is supported by a humid gaseous stream containing the hydrogen sulfide (Paulionytė & Vaiškūnaitė, 2023).

An ideal packing material for a pilot-scale biofilter should possess minimal bulk density to prevent sulfur accumulation (clogging) over time, maintain pH levels according to the activity of sulfur-oxidizing bacteria (SOBs) demand, is crucial for the desulfurization process of hydrogen sulfide, and optimize electrical conductivity to enhance chemical interactions and promote the conversion of H_2S into sulfate and sulfide compounds (Jia et al., 2022). This study aims to evaluate the most environmentally friendly organic/inorganic/synthetic packaging materials – to analyses their physical and chemical properties for effectiveness, to analyses their compatibility with the most advanced research technologies for removing hydrogen sulfide from biogas.

2. Sewage sludge and biochar

The utilization of biochar in the anaerobic digester is regarded as a valuable system for sewage management and the recovery of sulfur to enhance soil fertility (Bahraminia et al., 2020). Biochar is produced using pyrolysis, that is, biomass is super-heated in the absence of oxy-gen at high temperatures (350–700 °C) (Huan et al., 2021). The adsorption and oxidation of H_2S on biochar are believed to be facilitated by the presence of oxygen functional groups such as carboxylic and hydroxyl radical groups (Zeng et al., 2019). Studies on biochar substrates derived from sewage sludge, anaerobically digested fibers, and agricultural waste emphasize the importance of alkaline surface in H_2S removal, as the alkaline nature is believed to increase breaking H_2S structure process for further desulfurization reactions

(Huan et al., 2021). Biochar could serve as a cost-effective removal solution since it can be produced from various raw waste materials (Bahraminia et al., 2020). The results of implementing biochar samples as packing material in two different experiments are presented in Table 1 below.

Table 1. The capability of biochar samples utilized as packing material in various bioreactors

Packing bed	Biochar	Biochar
Pollutant	H_2S	H_2S
H_2S amount	105–1020 ppm	39 g m^{-3} h^{-1}
Time (day)	20	110
EBRT (s)	80	80
RE (%)	98	90
EC_{max} (g m^{-3} h^{-1})	94	90
Experiment properties	Bench-scale continuous-stirred tank reactor, municipal solid waste	Laboratory scale, Michaelis-Menten model
References	(Pudi et al., 2022)	(Pudi et al., 2022)

3. CLC waste and PUF

Cellular concrete waste may exhibit an H_2S removal performance of up to 32 (g m^{-3} h^{-1}) (Vaiškūnaitė, 2020). The complex physicochemical interactions between H_2S and various components of cellular concrete (primarily calcium oxide CaO from calcium silicate hydrate CaO SiO_2 nH_2O and ferric oxide Fe_2O_3) are responsible for these chemical interactions (Aryal et al., 2022; Das et al., 2022). These interactions result in the transformation of the material's structure into calcium sulfate (gypsum $CaSO_4$ $2H_2O$) and the formation of elemental sulfur (Das et al., 2022). Table 2 illustrates the outcomes of employing cellular concrete waste samples as packing material in an experiment.

The utilization of synthetic materials as packing materials in the biofiltration of H_2S from biogas represents a novel technique that has garnered significant attention from researchers as a clever approach to simultaneously address the issue of plastic waste management, which is often abandoned or discarded in the environment after use (Khanongnuch, 2019). In some cases, these plastics can persist in the environment for years, leading to significant environmental problems for wildlife and ecosystems (Pepper & Brusseau, 2019). Ideally, by advancing new technologies and developing alternative methods to reduce their impact on the environment, these unwanted chemical substances can be removed and repurposed for sustainable use (Lebrun et al., 2019). Polyurethane foam (PUF) materials have been found to possess the following properties: 1) high porosity facilitates increased contact between hydrogen sulfide and packing materials; 2) high specific surface area, high wettability, and nutrient capacity promote the growth of SOB (Sulfur Oxidizing Bacteria); 3) water retention and drainage capacity are beneficial for cleaning out reactor by-products. Compared to other materials, open-pore synthetic foams offer several advantages, including low density, high specific surface area, high porosity, and reasonable compaction resistance (Mamet et al., 2021). The outcomes of employing polypropylene samples as packing material in various experimental conditions are presented in Table 3 below.

Table 2. The capability of CLC waste example utilized in biofilter

Packing bed	Cellular concrete waste
Pollutant	H_2S
H_2S amount (ppm)	100 ppm
Time (day)	N/A
EBRT (s)	63
RE (%)	97
EC_{max} (g m^{-3} h^{-1})	5,6
Experiment properties	Laboratory-scale biofilter was packed with polyurethane foam
Reference	(Pudi et al., 2022)

Table 3. The capability of polypropylene, polyurethane, and polyethylene samples utilized in various bioreactors

Packing bed	Polyurethane foam	Polyurethane pall rings	Polyurethane foam
Pollutant	H_2S	H_2S	H_2S
H_2S amount	96 ppm	2000 ppm	4100–7900 ppm
Time (day)	78	365	119
EBRT (s)	40	60	N/A
RE (%)	98	98	99
EC_{max} (g m^{-3} h^{-1})	16,8	125	94,7
Experiment properties	Aerobic, Mesophilic microbial gathered from soils and sediments of a lake	Anoxic, sludge from wastewater treatment plant	Anoxic, pH 6.8–7.4, wastewater treatment plant
Reference	(Das et al., 2022)	(Das et al., 2022)	(Zeng et al., 2019)

4. Materials and methods

This research work was used dried sewage sludge from the Vilnius sewage treatment plant. Before, in this wastewater treatment plant, the sludge was dried at temperatures below 100 °C. Later, in the laboratory of Department of Environmental Protection and Water Engineering of Vilnius Gediminas Technical University, the dried sludge was pyrolyzed and biochar was obtained at three different temperatures (400 °C, 500 °C and 600 °C).

First, biochar samples were prepared into different fractions to better evaluate the physicochemical properties of this packaging material. Subsequently, every one of the four samples (sewage sludge, biochar after 400 °C, after 500 °C, and after 600 °C pyrolysis) were separated into two different size range of 0.6–1 mm and smaller than 0.6 mm. Consequently, in this research work, important physicochemical properties of two sewage sludge cases plus six unique biochar samples (in various sizes and temperatures) will be assessed.

Polyurethane foam is a latent material characterized by its low density, high porosity, significant expansion potential, and remarkably low cost. In the current study, polyurethane foam samples with a solid-state volume of 1 cm^3 were used as channel-packed beds. The surface area of this packing material is approximately 600 m^2/m^3.

Lightweight cellular concrete (CLC) is one of the most popular and widely used building materials, known for its durability and versatility. The primary differences between conventional concrete and CLC lie in the materials used, their physical properties, and their intended applications. The density of CLC is significantly lower than that of regular concrete, typically ranging from 400–1000 kg/m^3 compared to 2400 kg/m^3 for conventional concrete (Perez et al., 2020). Foam replaces the stone particles used in traditional concrete in the production of foamed concrete, with key components replaced by concrete, sand, foam, and water. The CLC waste sample used in this study has a median size of 11 mm.

4.1. Bulk density

A 100 ml glass chamber was filled with crushed biochar and dried for eight hours at 80 °C in a drying cabinet to determine the density. Subsequently, the chamber was shaken briefly to compact the dried samples and fill all the available spaces in the chamber (Strohmaier et al., 2019). Ultimately, division of each sample's mass (kg) to that sample's volume (m^3) will present as its bulk density value.

4.2. pH

To determine the average pH of each sample, 5 g of each packing material at various conditions was taken and mixed with 100 ml of deionized water. After allowing all mixtures to settle for 2–3 minutes to achieve a uniform solution, all sample combinations were placed on a shaker and left in a shaker at approximately 50 rpm for a duration of 60 minutes (Vaiškūnaitė, 2020). Eventually, all samples were filtered using filters with a porosity from 5–10 μm (Vaiškūnaitė, 2020; Jia et al., 2020). Figure 1 illustrates the arrangement of packing material samples and deionized water just beside paper filters.

Finally, the water obtained from filtering all packing material samples was individually analyzed using a pH meter (Jiao et al., 2022; Gao et al., 2022). Figure 2 displays a pH-meter with an accuracy of up to 0.001.

4.3. Electrical conductivity

After examining the electrical conductivity of all packaging materials using a probe and meter (Figure 3), the aim was to determine the electrical conductivity of each packaging material (Abd & Othman, 2022; Moradi et al., 2020). This result will estimate the amount of heavy metals present and their influence on the electrical conductivity of each packaging material analyzed.

Figure 1. During the experiments, the packaging material was filtered using deionized water and paper filters

Figure 2. The pH analysis of filtered packing materials

Figure 3. Evaluation of electrical conductivity of selected packing materials

5. Results and discussions

This section of the work is dedicated to the examination results obtained from precise scientific measurements conducted on selected packing materials (sewage sludge, biochar after pyrolysis at 400 °C, 500 °C, and 600 °C, cellular concrete (CLC) waste, and polyurethane foam (PUF)). As outlined in the Methodology section, the most significant physicochemical properties of the selected packing materials were bulk density, pH, and electrical conductivity. The outcomes of these examinations will offer a clearer perspective on how each of these properties can influence the desulfurization process of biogas from hydrogen sulfide.

5.1. Bulk density

Taking into account the weight of the chamber, which is 10.58 g (V = 10.58 g), the estimated bulk density for each sample is as follows, displayed in Table 4.

Firstly, it is evident that by increasing the size of biochar particles from below 0.6 mm to 0.6–1 mm, the bulk density of the represented sample significantly decreased. This decrease is likely due to the larger spaces available in the chamber for biochar samples with larger particles, which become more filled when the chamber is filled with smaller particles, allowing for more particles to be accommodated.

Table 4. Evaluated bulk density of selected packing materials

	Bulk density	
Packing materials	>0.6 mm	0.6–1 mm
Polyurethane foam (PUF)	30 kg/m^3	
Cellular concrete (CLC) waste	547 kg/m^3	
Sewage sludge	73 kg/m^3	55 kg/m^3
Biochar after 400 °C	79 kg/m^3	57 kg/m^3
Biochar after 500 °C	80 kg/m^3	58 kg/m^3
Biochar after 600 °C	80 kg/m^3	59 kg/m^3

Another point to consider is that as the pyrolysis temperature increased, the bulk density of both small and large particle samples also increased. This is because at higher temperatures, biochar particles become more compacted, moisture content decreases, and they can occupy less space than they used to. Regarding polyurethane foam (PUF), its low density benefits construction and reduces compaction issues with this type of packing material, as it exhibited the lowest density (30 kg/m^3) compared to the other evaluated materials.

5.2. pH

From the results presented in Table 5, it can be summarized that, in terms of biochar particle sizes, samples with sizes of 0.6–1 mm generally exhibited higher pH values compared to the same pyrolysis samples with sizes smaller than 0.6 mm.

Table 5. Evaluated pH of selected packing materials

	pH	
Packing materials	>0.6 mm	0.6–1 mm
Polyurethane foam (PUF)	5	
Cellular concrete (CLC) waste	9	
Sewage sludge	7.25	7.13
Biochar after 400 °C	7.32	7.18
Biochar after 500 °C	7.46	7.2
Biochar after 600 °C	8.89	8.46

This trend can be explained by the fact that as the specific surface area of the sample increases and particles have a greater chance to engage in chemical interactions, they tend to shift from alkaline to acidic nature. Additionally, when the pyrolysis temperature is increased, the overall pH of the sample is affected, with particles at higher temperatures showing a tendency to be more alkaline than acidic. Furthermore, as the pyrolysis temperature of biochar samples increases, the total pH difference (gap) between the represented samples in different sizes becomes larger and larger. This could be attributed to the nature of biochar, where at higher temperatures, the effect of particle size becomes negligible compared to the surrounding temperature.

5.3. Electrical conductivity

The measurement was conducted at room temperature of 21 °C, and the results presented in Table 6 are expressed in units of MicroSiemens per centimeter (µS/cm). Figure 3 shows the device used to analyze the rate of electrical conductivity for each biochar sample, with an accuracy of 0.1 µS/cm. Ultimately, the measurement concluded with the following data:

Table 6. Evaluated electrical conductivity of selected packing materials

	Electrical conductivity	
Packing materials	>0.6 mm	0.6–1 mm
Polyurethane foam (PUF)	283 µS/cm	
Cellular concrete (CLC) waste	N/A	
Sewage sludge	983 µS/cm	702 µS/cm
Biochar after 400 °C	225 µS/cm	191.2 µS/cm
Biochar after 500 °C	193.4 µS/cm	187.1 µS/cm
Biochar after 600 °C	187.4 µS/cm	185.9 µS/cm

The obtained results prove that under the same pyrolysis conditions, biochar particles with smaller sizes exhibit significantly higher electrical conductivity compared to those with larger sizes. This observation can be related to the fact that particles with smaller sizes have a larger surface area, allowing them more opportunities for exchange and interaction with external particles. Additionally, as the pyrolysis temperature increases for biochar particles, the corresponding samples' electrical conductivity decreases significantly. This phenomenon can be described and expected because most active chemical compounds involved in chemical interactions, resulting in electrical conductivity, evaporate and are removed due to the elevated temperature and leakage from the biochar particles (such as metal compounds). As the pyrolysis temperature increases, the difference in electrical conductivity between biochar samples with sizes of 0.6–1 mm and > 0.6 mm becomes closer. This trend can be anticipated since at high temperatures, the impact of particle's size becomes less significant as chemical interactions decrease. Since concrete is known to be a poor conductor of electricity, this parameter is not analyzed for this material. However, the polyurethane foam (PUF) sample exhibited better electrical conductivity compared to all biochar samples after pyrolysis, although it still has significantly lower conductivity than sewage sludge.

6. Conclusions

1. To prevent clogging of the packing materials inside the biofilter, the lowest possible bulk density should be chosen, which was observed for polyurethane foam (PUF) (30 kg/m^3), while the highest was identified for cellular concrete (CLC) waste (547 kg/m^3). As expected, as the size of biochar particles decreases, the density increases, suggesting that implementing particles larger than 0.6 mm and smaller than 1 mm is preferable based on this criterion.
2. In terms of microbial activity, since the microorganisms utilized in this work would be a community of aerobic and anaerobic sulfur oxidizing bacteria, which prefer a more alkaline environment, cellular concrete (CLC) waste and biochar after pyrolysis at 600 °C are the most suitable options (both close to pH 9), while polyurethane foam (PUF) (pH 5) is not an ideal packing material. It is worth noting that the pH of biochar particles continues to increase with the pyrolysis temperature.
3. Electrical conductivity is directly related to the presence of heavy metals. The results showed that sewage sludge exhibited the highest conductivity (983–702 μS/cm), but as the temperature increases, heavy metals are leached from the biochar samples, resulting in a decrease in conductivity, reaching approximately 186 μS/cm for biochar after pyrolysis at 600 °C, which is the lowest among the case studies. However, this parameter cannot be determined for cellular concrete (CLC) waste, as it is not conductive.

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EKSPERIMENTINIS PASIRINKTOS BIOFILTRACIJOS MEDŽIAGOS TYRIMAS DINAMINĖMIS SĄLYGOMIS

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Santrauka

Vandenilio sulfidas (H_2S) yra vienas iš nepageidaujamų toksiškų cheminių junginių, esančių biodujose, todėl jį reikia pašalinti. Šių dujų biofiltravimas yra aplinkai nekenksmingas būdas. Įprastą biofiltrą sudaro filtravimo medžiaga kaip sieros oksiduojančių bakterijų gyvenamoji vieta. Siekiant optimizuoti H_2S pašalinimo efektyvumą, eksperimentų metu turi būti įvertinta pasirinktų organinių filtravimo medžiagų fizikinių ir cheminių savybių įtaka biofiltravimo procesui. Tyrimo metu buvo nagrinėtos svarbiausios nuotekų dumblo mėginių charakteristikos ir bioanglis (po pirolizės: 400 °C, 500 °C ir 600 °C), taip pat akytojo betono atliekos ir poliuretano putos. Filtravimo medžiagų pasirinktos frakcijos buvo: mažesnės nei 0,6 mm ir nuo 0,6 mm iki 1 mm. Atlikti matavimai apėmė tūrinį tankį, pH ir elektrinį laidumą bei buvo aptartas šių savybių poveikis H_2S valymui iš biodujų.

Reikšminiai žodžiai: H_2S šalinimas, biofiltravimas, pakavimo medžiagos, fizikinės ir cheminės savybės.