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ACETONE, XYLENE AND AMMONIA REMOVAL ENHANCEMENT IN THE BIOFILTER PACKED WITH STEAM MODIFIED BIOCHAR

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Highlights

- Xylene is less removed in the self-irrigated biofilter due to limited mass transfer.
- Biochar modified with the steam can enhance removal of VOCs and ammonia.
- Results showed that biofilter system can remove ammonia up to 96%.

Abstract. The present study evaluated short-time effect of steam modified biochar with rhamnolipid solution on the removal of different volatile organic and inorganic compounds (acetone, xylene, ammonia) in biofilter with capillary system. Modification of pine wood biochar with steam governed better treatment of pollar acetone and ammonia compounds from the air stream with removal efficiencies of 93% and 96%, respectively. It was related to biochar's higher average capillary radius, moisture content and lower air velocity. However, removal of xylene was much lower and reached 72% in the case of modified biochar. It can be explained by its higher Henry's constant, hydrophobicity and non-polarity.

Keywords: gas-phase biofiltration, biochar, air cleaning technologies, steam, rhamnolipid solution, volatile organic compounds, ammonia.

Introduction

Volatile organic compounds (VOCs) are the main air pollutants. VOCs are frequent air pollutants in non-industrial environments (Molhave & Nielsen, 1992). VOCs have become significant pollutants due to their effects both on human health and atmospheric chemistry (Yalcin et al., 2020). They are emitted from various industrial companies and can affect human health and environment due to their toxicity and stability (Yousefinejad et al., 2019). Air pollution is one of main factors of premature deaths and diseases, and it causes the highest risk to human health in Europe, which accounts for 400,000 premature deaths per year in 39 European countries (European Environment Agency, 2019). VOC sources consist of vehicle exhaust gases, compounds valorisation from fuel storage tanks and gas distribution stations, ink printing, engine cleaning, paving roads with asphalt and others (Breton et al., 2017). In urban areas the main sources of VOCs emissions are anthropogenic. According to the European Water Framework Directive, VOCs are assigned to important pollutants which can cause hazard to human health and ecological condition of the environment (Cheng et al., 2016). Though overall VOC emissions are still relatively low, total solvent emissions for the EU were 1981 thousand tonnes per year in 2015 (Pearson, 2019). Main solvents are acetone, methanol, ethanol, isopropyl alcohol. It was found that even small VOC emissions can impact human health and cause nausea, eye and throat irritation, asthma induction, fatigue, dizziness and confusion (Masekameni et al., 2019). It has been proved that many VOCs are toxic. At high exposure levels VOCs are known to induce neurotoxic, carcinogenic, teratogenic and other adverse health effects (Molhave & Nielsen, 1992). It has been showed that some VOCs induce cancer to animals, and some of them are known to cause cancer to humans (Garcia-Jares et al., 2012). Wei et al. (2011) showed that xylene induces pathological changes in the mouse ovarian tissue. Exposure to

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VOCs results in cellular toxicity across the nervous and respiratory systems (Sakunkoo et al., 2021). VOCs threaten urban air quality and regional climatic changes through the formation of ozone and secondary organic aerosols (Borbon et al., 2013). These xenobiotic pollutants compose 7% of all atmospheric pollutants which are gradually increasing since the last century (Cheng et al., 2016). Except for VOCs, volatile inorganic compounds (such as ammonia) are also interesting pollutants used in biofiltration, since both are biodegradable in the aerobic way. Ammonia gained much attention as it can be easily identified from other composting odors as it is highly odorous gas and can be released in large amounts (Kavyashree et al., 2015).

Acetone is one of the most common VOCs and can cause serious air pollution and affect human health (Mo et al., 2019). Though acetone is genotoxic, majority of conducted genotoxicity studies were negative (Alberta Environment, 2004a). Short-time effect with acetone vapour can irritate human throat, nose and lungs, unconsciousness, dizziness, confusion, headache, tension, fatigue, irritability, general weakness, lack of energy, etc. The impact of acetone on humans usually causes risk to health via long-term contact (Cui et al., 2019). Anthropogenic acetone sources are vehicle exhaust gases, solvent usage and hydrocarbons oxidation (Guo et al., 2013). Other acetone sources are biomass (wood) combustion and oceans (Khan et al., 2015), pulp production, plastic incineration, municipal solid waste incineration, tobacco smoke, oil extraction and landfills. Global acetone emissions are 40–60 million tons per year (Alberta Environment, 2004a). Secondary acetone formation governs 50% of these emissions. Biomass combustion, direct biogenic emissions and primary anthropogenic emissions contribute to 26%, 21% and 3% of global acetone emissions, respectively. Biomass combustion is considered as the main source of acetone (Atherton, 1997).

Xylene is aromatic hydrocarbon and usually used in industry and medical technologies as a solvent (Kandyala et al., 2010). It is used as a solvent in the manufacturing of chemical materials (polymers and others), agricultural sprays, adhesives and coatings and low amounts of it are found in aviation fuel (World Health Organization, 2003). It is found in natural crude oil and natural gas (Alberta Environment, 2004b). Its emissions are formed during the incineration of organic materials (forests, grass and other biomass). Sources of xylene emissions include oil refineries, fuel terminals, chemical and polyester manufacturing industries, paints, dyes and varnishes (Reyna & Lee, 2005). Additionally, xylene is found in volcanic gas. Xylene is very reactive and contributes to the formation of ozone (O_3) and, at the same time, to the formation of photochemical smog (Bang et al., 2017). The principal contact of xylene with humans occurs through soil contamination, when pollutants leak from underground oil storage facilities. It can leak into the soil, surface and underground water bodies and to remain there unchanged for several or more months until it is decomposed into other chemical compounds (Kandyala et al., 2010). Short-term exposure to xylene governs irritation of the nose, throat and eyes, frequent headaches, dizziness, impairment of memory and reaction rate (Assessment report, 2004). China alone produced 1,161 million tons of xylene during 2015 (Khan et al., 2018).

Ammonia emissions are produced as a by-product from industrial processes, such as sewage sludge treatment, composting, livestock farming, agriculture (usage of mineral fertilisers) and petrochemical refining (Tsang et al., 2017). Livestock farming governs more than 70% of ammonia emissions in Europe, from which 50% come from cattle excrement, 30% from pigs and 20% from poultry litter (United Nations Economic Commission for Europe [UNECE], 2019). Usage of mineral fertilisers in agriculture governs 20% of ammonia emissions. Meanwhile, transport and industry account for only 10% of total ammonia emissions. Every year, the European Union (EU) generates 3.1 million tons of ammonia emissions (Zeng et al., 2018). It causes unpleasant odours, headaches, bronchial irritation and a burning sensation in eyes and skin (Tsang et al., 2017). Ammonia emissions from agriculture in EU is very high and reach up to 92% (Murawska & Prus, 2021). It is currently proposed to reduce ammonia emissions by limiting the use of urea fertilizers, replacing the use of mineral fertilizers with manure and adapting air scrubbers in areas of large livestock farms to protect the human health and environment (UNECE, 2019).

To sum up, VOC emission and odour removal technologies can be divided into physical, chemical or biological methods. Pollutant concentration, solubility and biodegradability are important parameters of biological treatment. Although there are different bioreactors (e.g. biofilters, drip filters, bioscrubbers), traditional biofilters are still the most commonly used biological treatment technology due to their relative simplicity, low investment costs and extensive experience over 10 years (Estrada et al., 2013). Biofiltration is a cost-effective and sustainable solution for the treatment of VOCs in the exhaust gases of industrial and waste treatment plants (Bruneel et al., 2018). Compared to other physical-chemical methods, such as adsorption, chemical oxidation, precipitation adsorption technologies, biofiltration is more thermodynamically efficient, environmentally friendly, economically efficient and socially acceptable (Lebrero et al., 2014). Biofiltration has been successfully used to control odours and remove organic and inorganic pollutants from stationary air sources (Kavyashree et al., 2015). Biofiltration has an advantage over other purification technologies that many organic and inorganic compounds are converted into harmless oxidation products. It is suitable for cleaning air with low concentration pollutants (Yousefinejad et al., 2019). In most cases biofilters are suitable for cleaning gases containing organic carbon compounds in concentrations less than 500 mg/m^3 (Colon et al., 2009). Acetone background concentration is reported to be in a range from 0.2 ppb in the southern hemisphere to 0.5 ppb in northern hemisphere (Khan et al., 2015). Average ammonia concentrations range from <1 ppbv in remote continental and oceanic areas >24 ppbv, typically over regions of intensive agriculture (Nair & Yu, 2020). In the US xylene average concentration in indoor and outdoor air environment is ranging from 1–10 ppb and 1–30 ppb (Niaz et al., 2015).

The biofiltration process is based on the passage of contaminated air through a biofilm of microorganisms that is immobilised in a porous material (Kavyashree et al., 2015). Contaminants diffuse from the gas into a thin layer of biofilm adhering to the porous material and are metabolised. The end products of complete biodegradation (microbiological oxidation) are CO_2, H_2O, microbiological biomass and mineral salts. In the case of ammonia, microorganisms decompose the compound into harmless NO_2^- and NO_3^- by-products. The growth of microorganisms' cells requires oxygen, organic and inorganic nutrients. Those nutrients should be in a dissolved, readily accessible form. Important nutrients are nitrogen and phosphorus salts. Bioagents that accelerate the biodegradation of pollutants in the biofilter include microscopic bacteria, yeast and fungi. Microbial consortium that is located on the organic matter of a substance uses pollutants as a source of carbon for their growth and supply. The mass transfer of hydrophilic compounds to the biofilm is efficient due to the small Henry's Law constants (H), while hydrophobic compounds (with a high H) have a limited mass transfer due to the resistance of the water layer surrounding the biofilm. Microorganisms can produce natural biologically active substances that can increase the solubility of VOCs in the aqueous layer and, therefore, improve mass transfer to the biofilm (Bruneel et al., 2018). Surface-active biomolecules (biosurfactants) produced by microorganisms can improve hydrophobic VOC's bioavailability (Dewidar & Sorail, 2022). Previous studies showed that rhamnolipids produced by *Pseudomonas aeruginosa* which are biosurfactants have potential efficiency in many environmental bioremediation applications, including biofiltration. Surfactants are known modifying the cell surface hydrophobicity by increasing hydrophobic functional groups. As a result it can increase hydrophobic VOC's affinity to the substrate due to reduction of aqueous surface tension and formation of micelles. Bacteria are the dominant microorganisms in the removal of VOCs, and high moisture content and neutral pH are favourable conditions for their development (Revah et al., 2011). In summary, bacteria are better suited for the removal of hydrophilic compounds under optimal conditions (Ghasemi et al., 2020). Bacteria *Pseudomonas* sp. are one of the bacterial species that is continuously formed in the biofilter biofilm during the removal of VOC. Meanwhile, fungi can absorb hydrophobic compounds faster than bacterial biofilms.

Recent studies have shown that fungi are particularly suitable for gases purification from hydrophobic compounds. It is hypothesised that the large area of hyphae promotes the absorption and transport of hydrophobic compounds from the contaminated gas stream to the cell surface (Kennes & Veiga, 2004). Fungi can effectively remove various impurities from the gas stream, thereby facilitating the mass transfer of hydrophobic compounds to aqueous liquids (Gospodarek et al., 2019). Fungi in biofilters have other advantages: enzymatic diversity, ability to grow in stressful environmental conditions such as limited nutrient availability, activity in low humidity and pH conditions, where bacterial growth is limited (Revah et al., 2011). Previous study (Baltrėnas & Mačaitis, 2014) found that at the beginning of the experiment (up to 7 days) micromycetes predominated among microorganisms and from the 10th day bacteria began dominating.

One of most investigated bio-surfactants, rhamnolipids, are mainly produced from Pseudomonas aeruginosa and effectively used in bioremediation of various pollutants (Zhou et al., 2019). They are formed from one or two rhamnose units linked to one or two chains of fatty acids. It is effective in reducing water surface tension (from 72 mN/m to 3 mN/m), as well as oil-water interfacial tension. The most important benefits of biosurfactants are their ecological compatibility, low toxicity and biodegradable nature. Rhamnolipids are expected to be more effective than synthetic surfactants. Rhamnolipids have been used in many fields such as bioremediation of hydrocarbons from contaminated soil, heavy metal removal, soil treatment (Costa et al., 2010). Recent study (Dewidal & Sorial, 2022) showed that application of rhamnolipids in fungal-cultured biotrickling filter had a significant effect on toluene removal. Removal efficiency reached 96% in a biotrickling filter fed with 150 mg/l rhamnolipids.

The quality of filter material is considered to be one of the key factors for the efficient operation of the biofilter (Kavyashree et al., 2015). In biofilters, a contaminated air stream or odorous gas is passed through a biologically enriched filter material, such as soil, wood sawdust, compost or mixed materials that biodegrade adsorbed/absorbed contaminants (Pagans et al., 2005). Packing materials used in biofilters for air treatment can be: pine wood bark (Andres et al., 2007), granular mineralized and fibrous peat (Alvarez-Hornos et al., 2007), coconut fiber, peat, compost from wastewater treatment plant and pine leaves (Maestre et al., 2005), a mixture of vermicomost and wood charcoal (Ghasemi et al., 2020). This study evaluated the efficiency of pine wood biochar modified with the steam of rhamnolipid solution in the removal of volatile organic (acetone, xylene) and inorganic (ammonia) compounds. The benefits of biochar modification with steam is that it increases biochar's surface area, microporous surface area, hydrophilicity, creates hydroxyl, carboxyl and carbonyl functional groups. High specific surface area and water retention are principal characteristics of pine wood biochar modified with the steam of rhamnolipid solution that can make it suitable for the removal of VOCs and ammonia. High surface area is needed in order to achieve high mass-transfer velocities; high water retentivity is needed in order to keep optimal activity of the immobilized microorganisms

(Dorado et al., 2010). The objectives of this study were: 1) to modify low-temperature (450 °C) pine wood biochar with the steam of rhamnolipid solution; 2) to evaluate effect of biochar modification with steam on the removal efficiency of acetone, xylene and ammonia. The aim of this research was to evaluate the effect of steam modified pine wood biochar with rhamnolipid solution on the removal efficiency of different polarity volatile organic and inorganic compounds (acetone, xylene and ammonia) in the biofilter with self-regulating irrigation system. Previous studies showed that usage of rhamnolipids in biological air treatment devices can significantly improve removal of volatile orgnanic compounds from the air stream.

1. Material and methods

1.1. Volatile organic and inorganic compounds

Three volatile compounds were selected grounding on their different physicochemical properties (Table 1). Based on Henry's constants (from $1.61\cdot10^{-5}$ until $7.18\cdot10^{-3}$), this study evaluated the biodegradation of different wettability compounds (from hydrophilic in the case of acetone and ammonia to moderately hydrophobic in the case of xylene).

Table 1. Physico-chemical properties of acetone, xylene and ammonia, ND – no data (National Research Council, 2010; Bruneel et al., 2018)

Parameter	Unit	Acetone	Xylene	Ammonia
Chemical formula	–	C_3H_6O	C_8H_{10}	NH_3
Molecular weight	g/mol	58.1	106	17
Odor limit value	ppmv	100	1	46.8
Boiling point	°C	56.2	137–140	–33.3
Vapor pressure (at 20 °C)	mmHg	182	6.72	6661
pH	pH units	7	ND	11,6
Density	g/cm^3	0.78	0.86	0.68
Solubility in water (at 25 °C)	mg/l	100 000	130	895 000
Polarity	–	Polar	Non-polar	Polar
Topological polar surface area	Å^2	17.1	0	1
Henry's law constant (at 25 °C)	atm-m^3/mol	$1.87\cdot10^{-5}$	$7.18\cdot10^{-3}$	$1.61\cdot10^{-5}$

1.2. Structure and operation of the devices

Biofilter consisted of inlet and outlet ducts and a tank containing 86 plexiglass tubes of 0.3 m length and 0.02 m inner diameter, which were filled with wood biochar and fibre biocharge (Figure 1). In the first experiment, the tubes were filled with unmodified pine wood biochar (450 °C, 2 h) and wood fiber at ratio of 10 to 1 and, in the second experiment, with a pine wood biochar modified with a steam of rhamnolipid solution (for 1 h) and wood fibre at the same ratio. Biochar was produced in a muffle furnace (E5CK-T) and the biomass was wrapped in aluminium foil for making oxygen deficiency conditions (Baltrėnaitė et al., 2017). Birch fibres were obtained by heat treatment of birch sawdust in a steam reactor at a pressure of 32 bars and a temperature of 235C (Baltrėnas & Mačaitis, 2014). A biochar hydrophilicity enhancement device was used for the biochar modification (Figure 2). The purpose of this device is to enhance biochar hydrophilicity in order to improve its wettability by increasing biochar's internal surface. The device consists of chamber filled with rhamnolipid solution, valve which connects the chamber of the solution with a biochar vacuum treatment chamber, heating element, temperature sensor, automatic control block, perforated partition, electric motor, shaft with mixing blades, vacuum treatment chamer, vacuum pump and biochar particles. Biochar was modified by using the steam of rhamnolipid material, heated up to 100–105 °C and applied in vacuum of 99 990 Pa – 1333 Pa by the vacuum pump in the biochar treatment chamber. Steam from contaminated air flow was created by mixing concentrated solutions of acetone, xylene and ammonia with deionised water. The air flow rate in a biofilter was controlled using an inlet valve. A volumetric air flow rate up to 10.5 m^3/h was used throughout the experiment.

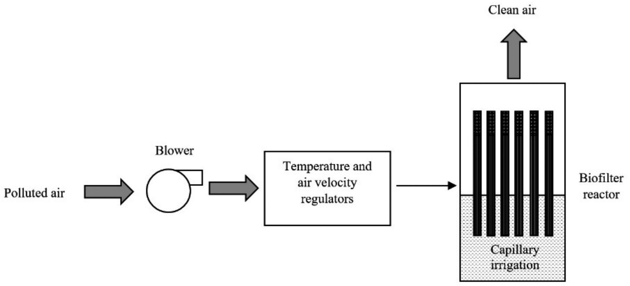

Figure 1. Scheme of a biofilter with a capillary system

Figure 2. Biochar hydrophilicity enhancement device (Baltrėnas et al., 2019)

1.3. Ensuring biofilter operating conditions (humidity, temperature and air velocity)

For the assurance of biocharge capillary irrigation, tubes were immersed in a nutrient solution up to a depth of 7.5 cm. The solution of mineral salts was prepared by dissolving K_2HPO_4 (1 g), KCl (0.5 g), $MgSO_4{\cdot}7H_2O$ (0.5 g), $FeSO_4{\cdot}7H_2O$ (0.1 g), $NaNO_3$ (0.9 g) in 1 L of water (Zagorskis et al., 2012). The relative humidity and air temperature near the biofilter were assessed using a humidity-meter M0290. The average room air temperature in both experiments was 21.5 °C and relative humidity was 54.6%. To maintain the required temperature for the biocharge in biofilter, the air stream was heated by a temperature controller installed in the gas inlet pipe (Figure 1). All experiments were maintained an average temperature of 33.1 °C in the biofilter inlet tube, 24 °C in the outlet tube and 26.6 °C in the tank. Temperature values were in the optimal range required for the removal of pollutants, from 15 to 35 °C (Pagans et al., 2005). Meanwhile, an average humidity of 53.6% was maintained in the biofilter inlet pipe, 95.8% in the outlet pipe and 61.9% in the tank. It is recommended to maintain 85–95% humidity in the biofilter (Baltrėnas et al., 2015b). The first study was performed at an average air flow rate of 0.37 m/s and the second at 0.32 m/s. The air flow was supplied to the unit using a fan that is installed in the air supply pipe. The air velocity in the biofilter was assessed using a Testo 400 instrument.

1.4. Analysis of biocharge and biogenic solution properties

Physico-chemical properties of biocharges after biofiltration were analysed to assess the moisture content and pH. Moisture content of biocharge was evaluated by the weighting method, which is based on a material's mass decrease after drying (Zagorskis et al., 2012). Lids with caps were dried at 105 °C for 1 h in a drying chamber and then cooled to room temperature before sampling. Dried bottles with caps were weighed on analytical balances. 1 g of a sample was taken with clippers and placed in bottles with caps. The sample was weighed and then dried in a drying chamber for 3 hours The moisture content was calculated according to the 1st formula (Brischke & Wegener, 2019):

$$MC = \frac{m_{wet} - m_{dry}}{m_{dry}} \times 100\%. \tag{1}$$

Biochar specific surface area was estimated by BET (Brunauer-Emmett-Teller) analysis (Ameloot et al., 2015) using a Nova 4200 E-series analyser (Quantachrome Instruments). Differences between unmodified and modified biochar's porous structure and surface morphology were assessed employing the SEM analysis (Gabhi et al., 2020). SEM is common direct method used for the separation of different pore size and for the measurement of pore size. The macrostructure of biochar was evaluated using an electron microscope. Scanning electron microscopy was performed using a field emission scanning electron microscope JEOL ISM-7600 F. Specifically, high-zoom photographs were used to estimate biochar pore diameters. "ImageJ" software with 2D pictures was used for the measurement of pore radius.

The pH of biogenic solution was assessed using a Mettler Toledo pH-meter (Baltrėnas & Mačaitis, 2014). During the study, the pH of biogenic solution reached 8.29±0.05 pH conditions required for the efficient gas cleaning in biological cleaning systems ranging from 6 to 8 for heterotrophic bacteria (Revah et al., 2011). The temperature of biogenic solution was recorded with an alcohol thermometer. In order to maintain a constant temperature of the biogenic solution of about 30 °C, a heating element was installed in the lower part of the biofilter tank. The mean temperature of the biogenic solution was 32 °C±1.

1.5. Monitoring of pollutant concentrations and evaluation of biofilter efficiency

After activation, 300 mg/m^3 of acetone vapour, 300 mg/m^3 of second pollutant (xylene) vapour and 300 mg/m^3 of third pollutant (ammonia) vapour were being fed to the biofilter for 5 days. The required pollutant concentrations were achieved by diluting the liquid phase pollutant with distilled water. Concentrations were determined with a photoionization detector, a MiniRAE 2000 instrument (*American company RaeSystems*) at eight points (for 5 min). The biofilter's air purification efficiency was calculated according to the 2nd formula:

$$E = \left(\frac{C_1 - C_2}{C_1}\right) \times 100\%,$$

where: E – biofilter cleaning efficiency (%); C_1 – pollutant concentration before cleaning (mg/m^3); C_2 – pollutant concentration after cleaning (mg/m^3). In addition to the removal efficiency, the parameters, such as volumetric load (LV) and elimination capacity (EL), were also evaluated (Baltrėnas et al., 2015b). The formulas for calculations are given below:

$$LV = \frac{Q \cdot C_I}{V}; \tag{3}$$

$$EC = \frac{Q \cdot (C_I - C_0)}{V}, \tag{4}$$

where: LV – volumetric load (mg/m^{3}h); V – reaction volume (m^3); Q – volumetric flow rate (m^3/h); EC – elimination capacity (mg/m^{3}h).

1.6. Microbiological activation and characterisation of biocharge

In order to maintain a high efficiency of air cleaning, high activity of microorganisms in the wet biocharge must be maintained (10^6–10^9 cfu/g) (Baltrėnas et al., 2015b). This time interval is called the activation time of microorganisms. The duration of biocharge activation for each experiment lasted for 2 weeks. The concentrations of the pollutants in the vapour that passed through the biocharge were about 20 mg/m^3. The contaminants were being fed to the unit four times a day for 15 min. It was then delivered every two days extending the delivery time of the pollutant up to 1 h. Visual differences were found between macroscopic fungal colonies in the case of different biocharges 7 days after the start of biofiltration.

2. Results and discussion

2.1. Physical-chemical properties of the biocharges

After biofiltration, it was found that the biocharge of unmodified biochar and fibre had a moisture content of 58.9%, and the biocharge of steam modified biochar and fibre had a moisture content up to 8% more (67%). The pH of biocharge was evaluated in 0.01 M $CaCl_2$ solution at ratio of 1 to 5 (European Biochar Certificate, 2012). The suspension was shaken for 1 h and then the pH was assessed using a Mettler Toledo Seven Multi pH-meter. The physicochemical properties of the materials used for biological air purification are given in Table 2. It can be seen that biochar modified with the steam of rhamnolipid solution, biochare's moisture content increased up to 8 percent units. Comparing pH of different biocharges, pH of modified biochar was up to 0.6 pH units higher.

Table 2. Physicochemical properties of biocharges used in biofilter after biofiltration, n = 3, ±standard deviation

Biocharge	Moisture content, %	pH
Mixture of unmodified pine wood biochar and birch fiber	58.9±2.13	5.5±0.06
Mixture of pine wood biochar modified by the steam of rhamnolipid solution and birch fiber	67±2.08	6.1±0.17

Each species of microorganism is most successful in living in a certain pH range and can be inhibited or killed if conditions fall outside the range. Species that tolerate moderate pH are probably the most common, therefore many biofilters are designed close to a pH range of 7. This is generally considered to be a favorable conditions for microorganisms' development and growth. The initial assumption that the selected contaminant is best purified at pH 7 is generally accepted as a conservative guess, but this is often not confirmed by actual data. In fact, some systems can operate successfully at a different pH. Biochar surfaces can carry a net negative or positive charge depending on pH of the solution. When pH > pHzpc, the biochar surface is negatively charged, and when pH < pHzpc, the surface carries a positive charge. This can enable electrostatic attraction between the biochar surface and the anionic or cationic species of pollutants. The degree of dissociation of organic pollutants is also pH dependent (Gwenzi et al., 2021).

The structure of tested biochar types is shown in Figures 3a, 3b. The average pore diameter of unmodified biochar was 17.5±2.68 μm and that of modified with the steam of rhamnolipid solution was 22±5.19 μm. The specific surface area of unmodified pine wood biochar was 3.22 m^2/g. According to other studies, surface area of pine wood biochar (450 °C, 1.5 hours) reached 9.16 m^2/g and surface area of birch wood biochar (450 °C, 1.5 hours) – 5.92 m^2/g (Baltrėnas et al., 2015a). The surface area was calculated form the adsorption isotherms using BET model. According to previous study (Usevičiūtė et al., 2021), for the steam-modified birch bark biochar, specific surface area reached 15.2 m^2/g.

Higher surface area of biocharge is related to better pollutant removal efficiency of VOCs in biofilter (Baltrėnas et al., 2015b). The greater the porosity of biocharge is, the more efficient pollutent degradation is. Literature review showed, that carbon materials have been successfully used in VOC adsorption for their porosity and high surface area (Zhang et al., 2017). One study (Rajapaksha et al., 2015) showed that steam activation significantly increased tea waste biochar's and burcucumebr biochar's surface area by 68 and 207%, respectively. It was attributed to enlargement of pore volumes. Shim et al. (2015) revealed that biochar made from Miscanthus sacchariflorus at 500 °C had much lower surface area compared to steam activated product (181 and 322 m^2/g, respectively). In physical activation, oxidizing gases such as steam are used to increase surface area of carbonaceous products by partial oxidation (Hagemann et al., 2020). Steam activation may be effective for creating new porosities and increasing surface area of biochars, which could significantly increase sorption capacities of VOCs. Baltrėnas et al. (2015b) used thermally treated birch wood, which had porous structure and bigger specific surface area for the removal of acetone in a biofilter with straight plates.

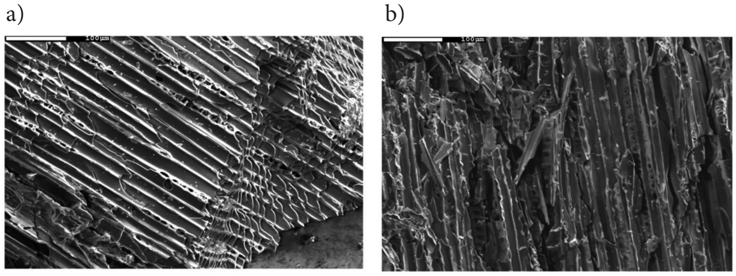

Figure 3. Structure of the studied biochar types: a) unmodified pine wood biochar; b) pine wood biochar modified by the steam of rhamnolipid solution (magnification 200 times)

2.2. Biofilter cleaning efficiency for acetone

According to Figure 4, a high efficiency of air purification from acetone can be achieved immediately 14 days after the bio-charge activation. From 1 to 5 days after activation, the efficiency of acetone vapour removal in the biofilter filled with unmodified biochar and fibre varied from 68.2 to 83.8% when the average air flow rate through the biocharge was 0.37 m/s. This can be attributed to the potential increase in the number of bacteria (Baltrėnas & Mačaitis, 2014). In this study, the initial acetone concentration ranged from 277 to 356 mg/m^3. The relative humidity was maintained at 57–91.6% during the experiment and the moisture content of the biochar was maintained at 57–61.8%. In the case of steam modified biochar, the removal efficiency was 78.2–92.8% over the period of 1–5 days, which was 4.53–15.1% more compared to a biocharge of unmodified biochar mixture on days 1–5 of the experiment, when the average air flow speed in the biofilter was 0.32 m/s. Initial acetone concentration ranged from 291 to 316 mg/m^3. It can be stated that biochar modification with a steam of rhamnolipid solution significantly (up to 9%) increased the efficiency of acetone purification in a biofilter with spontaneous microorganisms.

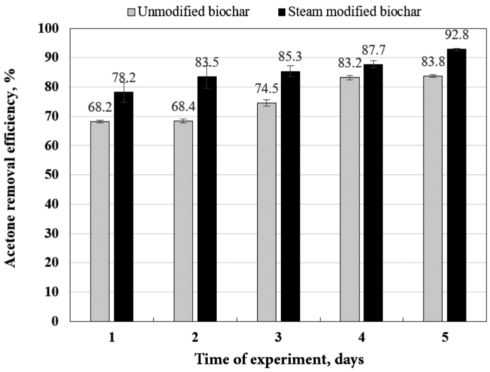

Figure 4. Pollutant cleaning efficiency dependence on time in biofilter filled with unmodified biochar and fiber biocharge to which air stream contaminated with acetone vapor was supplied (bars represent standard deviation, n = 3)

According to another study (Baltrėnas et al., 2015b), similar acetone cleaning efficiency, which ranged from 70 to 90% when a biofilter with straight structure plates and wood fibre biocharge was applied, was obtained. In the above mentioned study, the highest acetone removal efficiency (90.3%) was achieved when the acetone concentration was 300 mg/m^3.

2.3. Biofilter cleaning efficiency for xylene

The data from a biofilter experiment with unmodified biochar and fibre biocharge and xylene cleaning efficiency is shown in Figure 5. The efficiency of the removal of xylene from the air after 5 days when the activation period ended was found to be lower than that of acetone and reached 68.9% when an average air flow rate through the biocharge was 0.37 m/s. Initial xylene concentration in this study ranged from 314 to 371 mg/m^3. Poorer biodegradation of xylene compared to acetone or ammonia can be related to its higher Henry's constant (7.18·10^{-3} atm-m^3/mol), which indicates the level of gas solubility in water. Hydrophobic compounds (with high H) have limited mass transfer due to the resistance of the water layer surrounding the biofilm. Over a period of 1–5 days, the xylene removal efficiency ranged from 65.1 to 71.6% and was up to 8.5% higher in the case of steam modified biochar compared to the unmodified biochar. This can be attributed to better moisture retention conditions in the biofilter and better fungal development. Initial xylene concentration ranged from 268 to 347 mg/m^3.

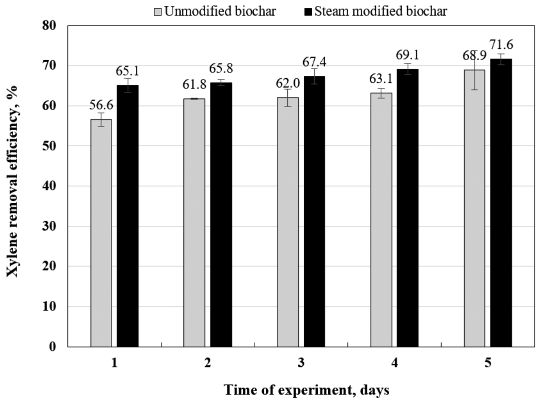

Figure 5. Pollutant cleaning efficiency dependence on time in biofilter filled with unmodified biochar and fiber biocharge to which air stream contaminated with xylene vapor was supplied (bars represent standard deviation, n = 3)

Efficient gas-liquid mass transfer is of great significance in many industrial applications. When mass transfer is limited, the metabolic rate of microorganisms decreases. Enhancmenet of mass transfer in biofilter can be icreased with power greater power consumption. A structure (packed-bed) is used in laminar contractors (biofilters) to maximize the contact surface, but the lack of mixing in these systems leads to the presence of heterogeneities within the packed-bed (Kraakman et al., 2011). New strategies to increase mass transfer in gas treatment operations while minimizing the power consumtion need to be developed. Besides, mass transfer can be improved if hydrophilic VOCs will be fed into the biofilter since high gas-liquid transfer rates make target pollutants more available to microorganisms. Other studies have also showed that the biodegradation of VOCs in biofilters is strongly influenced by the Henry's constants. Deshusses and Johnson (2000) studied 18 VOCs with different Henry's constants in a biofilter for 48 hours and concluded that the biodegradation of VOCs was strongly dependent on their availability or Henry's constants and hydrophobicity (octanol/water distribution). It was similarly found that contaminants with high Henry's constants are difficult to remove. The biodegradability of xylene is also limited by its toxicity (Morgado et al., 2004). In another study (Baltrėnas & Mačaitis, 2014), the maximum xylene removal efficiency was 87.9% in a biofilter with porous polymer plates, birch fibre biocharge and capillary irrigation system. Morgado et al. (2004) found that in the compared three studied materials (peat, wood sawdust and granular basalt rock (40–50% SiO_2)), peat had the best effect on the removal of m-xylene from the air by biofiltration.

Biological air purification from xylene occurs due to two phenomena: 1) physicochemical, which determined the mass transfer from gas to the liquid phase (biofilm), by which biomass develops; 2) biological, which is responsible for the oxidation of xylene by microorganisms (Morgado et al., 2004). Based on the removal of VOCs in the biofilters, there is the first dominant phase of the absorption process, during which the removal efficiency of the pollutants is lower due to the saturation of the absorption capacity and the adaptation period of microorganisms (Colon et al., 2009). Over time, the biofilter cleaning efficiency increases due to the dominance of biodegradation. This may explain a high removal efficiency during the during the last days.

2.4. Biofilter cleaning efficiency for ammonia

Figure 6 shows the time dependence of ammonia concentrations and its treatment efficiency in the biofilter filled with unmodified biochar and steam modified biochar. Biofilter filled with unmodified biochar cleaning efficiency significantly increased over 5 days of the experiment and ranged from 68.5 to 78.8%. In the case of unmodified biochar, the maximum removal efficiency was similar both for acetone and ammonia (83.8% and 78.8%, respectively). This could be explained by a similar water solubility coefficient ($1.87 \cdot 10^{-5}$ atm-m^3/mol and $1.61 \cdot 10^{-5}$ atm-m^3/mol, respectively). This pressure dependence is defined by Henry's law, which states that the solubility of gas in water is directly proportional to the pressure of that gas. On days 4th and 5th of the experiment, the ammonia removal efficiency was constant and reached 78.6 and 78.8%. Initial ammonia concentration ranged from 272 to 338 mg/m^3. The efficiency of the biofilter filled with a modified biochar and fibre biocharge was higher and ranged from 84.9 to 96.4%. It can be seen that, in the case of modified biochar, the efficiency of ammonia removal was much higher than in the case of unmodified (up to 17.6%). The initial ammonia concentration ranged from 264 to 273 mg/m^3.

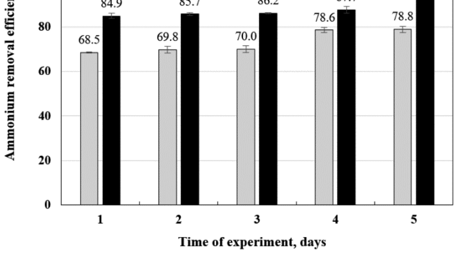

Figure 6. Pollutant cleaning efficiency dependence on time in biofilter filled with unmodified biochar and fiber biocharge to which air stream contaminated with ammonia vapor was supplied (bars represent standard deviation, n = 3)

One study (Zhang et al., 2008) obtained ammonia removal efficiency in the range of 80 to 100% in a hybrid biofilter with zeolite and poplar sawdust biocharge at various initial ammonia concentrations. The maximum biological removal capacity was 10 gN/m^{3}h. The optimal volumetric air flow rate was 0.7 m^3/h. According to Gabriel et al. (2007), the ammonia removal efficiency in the case of coconut fibre biocharge ranged from 30 to 100%. These authors claim that dry zones in biofilter result in lower ammonia removal efficiencies.

The main strategies for the removal of volatile pollutants in a biofilter are in principle similar to each other. The two main mechanisms of biofiltration are: 1) mass transfer from air to the water layer surrounding the biocharge due to adsorption and dissolution, and 2) biodegradation of sorbed contaminants in the biofilm. The mass transfer of hydrophilic compounds (such as acetone and ammonia) to the biofilm is efficient due to the low Henry's law constants, while hydrophobic compounds (such as xylene) with a high Henry's constant have limited mass transfer due to the water layer surrounding the biofilm.

2.5. Results of biofilter performance parameters

Volumetric load, elimination capacity and removal efficiency were evaluated on the relationship between input and output of pollutant concentration, air flow rate and effective biofilter volume. Based on Table 3, it can be stated that a biofilter with a charge of unmodified biochar and wood fibre, regardless of the removal efficiencies, had a better performance in the case of acetone and xylene due to obtained elimination capacities (81 625 and 75 413 mg/m^{3}h, respectively) compared to modified biochar and fibre biocharge. Meanwhile, in the case of ammonia, a biofilter filled with steam-modified biochar and fibre biocharge performed better (elimination capacity of 76 730 mg/m^{3}h). The volume flow rate (Q) for biofilter, filled with unmodified biochar, was 10.5 m^3/h and for biofilter, filled with steam modified biochar, was 9.04 m^3/h.

Table 3. Pollutants volumetric loading rate, elimination capacity and average removal efficiency for two different biocharges (± standard deviation)

Biocharge	Pollutant	Volumetric load (mg/m^{3}h)	Elimination capacity (mg/m^{3}h)	Removal efficiency (%)
Mixture of unmodified biochar and fiber	Acetone	107 699	81 625	75.6±0.68
	Xylene	121 013	75 413	62.5±2.02
	Ammonia	101 704	74 755	73.2±1.19
Mixture of steam modified biochar and fiber	Acetone	91 457	81 329	85.5±2.14
	Xylene	93 868	63 370	67.8±1.39
	Ammonia	87 056	76 730	88.2±1.06

A weak linear relationship was obtained between the pollutant volumetric load and elimination capacity for both biocharges, and the maximum removal capacity was obtained in the case of acetone when it reached 81,625 mg/m^{3}h at 107,699 mg/m^{3}h volumetric load. The 5th day average acetone removal efficiency was 75.6% for the unmodified biochar and 85.5% for steam modified biochar (Table 3). In addition to acetone, ammonia had a high elimination of 76 730 mg/m^{3}h at 87 056 mg/m^{3}h volumetric load. The 5th day average ammonia removal efficiency was 88.2%. In the case of ammonia, the best elimination capacity was obtained for the modified biochar and fibre biocharge. Further research is needed to increase long-term operation of self-regulating biofilter with capillary system. Limitations of the biofiltration system include: excess biomass accumulation, uneven distribution of biomass and nutrients, low mass transfer of hydrophobic VOCs from the gas phase to the biofilm, what results in a low biodegradation of these compounds.

2.6. Evaluation of microorganism colonies

In the case of both biocharges, fungal colonies dominated throughout the experiment (Figures 8a–d). Fungi in biofilters have certain advantages over bacteria: they are more resistant to acidic environment and dehydration and external fungal hyphae form a larger surface area compared to bacterial biofilm, which enhances the decomposition of hydrophobic compounds (Groenestijn et al., 2001). Assessing the influence of air flow on the development of microorganisms, it can be seen that it affects the development of fungi, as they were visually less near the air inlet area (fungal colonies occupied 52.6–55.8% of the outer area of the first three tubes), meanwhile the farthest tubes from the air inlet were the most occupied by the fungi (the colonies covered 73.7–78.9% of the outer area of the last three tubes) (Figure 7). Sand coloured powdery mould colonies appeared to have dominated in the case of unmodified biochar and fibre biocharge.

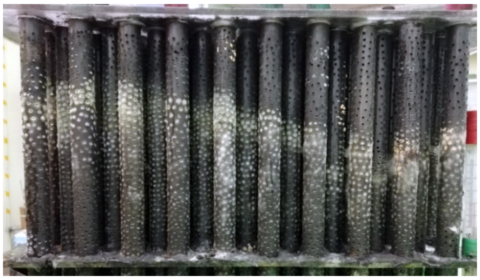

Figure 7. Macroscopic image of microorganism populations in a laboratory biofilter filled with a mixture of unmodified pine wood biochar and fiber on the 23rd day of the experiment

According to another study (Baltrėnas & Mačaitis, 2014), micromycetes of *Penicillium* sp., Aspergillus niger, *Aureobasidium* sp., *Geotrichum* sp. were predominant fungal species in the biocharge when the acetone vapour was passed through. In other study (Baltrėnas et al., 2015), spontaneous microorganisms (*Bacillus*, *Pseudomonas*, *Stapylococcus* ir *Rhodococcus*) developed and degraded acetone in the biofilter. Among the fungi, *Paecilomyces variotti* predominated and, among the yeasts, the genera *Aureobasidium* and *Geotrichum*, when xylene steam was purified (Baltrėnas & Mačaitis, 2014). In the case of ammonia, the dominant fungal species were Penicilium sp., white colonies forming *Myrothecium* sp., of which *Geotrichum* sp. and *Paecilomyces variotti* were dominant (Baltrėnas & Mačaitis, 2014).

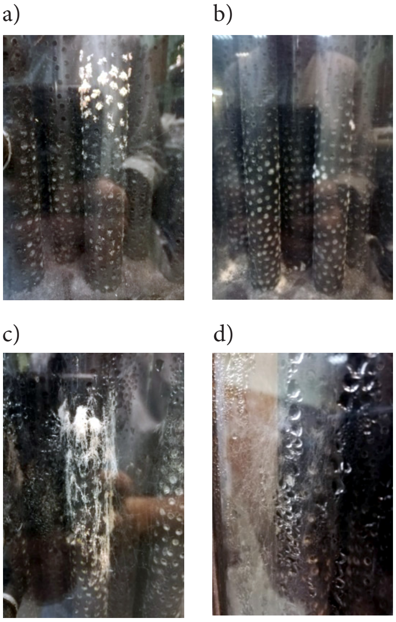

Figure 8. Macroscopic image of microorganism populations in a laboratory biofilter filled with a mixture of unmodified pine wood biochar and fiber (a–b) and a mixture of steam modified biochar and fiber (c–d) 7 days after the start of biofilter operation

Conclusions

1) Biofiltration technology using the self-regulating capillary process of steam modified biochar with rhamnolipid solution and wood fibers through capillary rise and development of spontaneous microorganisms has resulted in 9% and 17% higher removal of polar acetone and ammonia compounds, respectively. The removal efficiencies of acetone and ammonia for steam modified biochar were 93% and 96%, respectively. This can be attributed to higher relative humidity (up to 8.3%), higher biocharge moisture content (up to 9.7%) and lower average air velocity of 0.32 m/s. Water in a biofilter is essential for the growth/activity of microorganisms and nutrients transport.
2) The higher moisture retention of steam modified biochar could be due to the higher average radius of biochar capillaries (22 μm). When biochar capillary radius is larger, the capillary processes are faster. A capillary irrigation system in a biofilter is more economically efficient compared to artificial irrigation systems.

3) For both unmodified and modified biochar and fibre biocharges, xylene removal efficiencies were lower compared to acetone and ammonia which were 69% and 72%, respectively. This can be related to non-polarity of xylene and its higher Henry's constant ($7.18 \cdot 10^{-3}$ atm-m^3/mol) compared to other studied pollutants. Hydrophobic compounds (such as xylene) have limited mass transfer due to the resistance of water layer surrounding the biofilm.

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Declarations

- The authors have no relevant financial or non-financial interests to disclose.
- The authors have no conflicts of interest to declare that are relevant to the content of this article.
- All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.
- The authors have no financial or proprietary interests in any material discussed in this article.

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