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Active botanical biofilters for nitrogen dioxide and ozone removal using granular activated carbon

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ABSTRACT

Botanical biofilters can remediate numerous air pollutants and show potential for the removal of indoor NO_2 and O_3. Granular Activated Carbon (GAC) is a promising new addition to botanical biofilter growth media, increasing efficiency in Volatile Organic Compound (VOC) remediation, but it remains untested for other gaseous pollutants. This work assessed the capacity of an active botanical biofilter with a GAC growth medium to filter gaseous NO_2 and O_3 within a closed-loop flow-through reactor. We incorporate the effects associated with two plant species, *Spathiphyllum Wallisi* and *Syngonium Podophyllum*, substrate moisture, and varying ratios of GAC to coco coir on pollutant removal efficiency. All GAC containing substrates exhibited exponential decay for NO_2, with a 50% GAC wetted substrate composition producing the peak decay rate ($0.27 \pm 0.048\,ppb.s^{-1}$) and Clean Air Delivery Rate (CADR) at $1013.0 \pm 173.1\,m^3{\cdot}h^{-1}{\cdot}m^{-3}$of biofilter substrate. All treatments demonstrated non-significant removal of elevated O_3, possibly due to higher concentrations tested in the current work. There was no difference in NO_2 and O_3 removal rates or CADR between the two-plant species. This work provides promising results for the use of GAC within an active botanical biofilter to improve the removal of high concentrations of NO_2.

STATEMENT OF NOVELTY

Botanical biofilters have demonstrated promising *in-situ* results for reducing indoor and urban air pollutants. The current research highlights the air-cleaning potential a granular activated carbon-based growth substrate can add to botanical biofilters for the removal of NO_2, but not O_3. Under elevated NO_2 concentrations, a growth substrate composition of 50% GAC and coconut husk produced an average clean air delivery rate of $1013.0 \pm 173.1\,m^3\cdot h^{-1}\cdot m^{-3}$ of the biofilter substrate, outperforming botanical biofilters without GAC that underwent the same testing conditions. Furthermore, insight has been provided into the adsorption characteristics of GAC within botanical biofilters.

KEYWORDS

Green walls; indoor air quality; nitrogen dioxide; ozone; sustainability; sustainable infrastructure

Introduction

Conventional indoor environmental quality management systems for buildings indoor air quality and climate include heating, ventilation and air-conditioning (HVAC), functioning by flushing indoor environments with outdoor 'fresh air' which has been temperature modulated. While HVAC units are commonly equipped with air filters of a minimum efficiency reporting value (MERV) of 8–13 these are only effective for PM removal and are incapable of gaseous pollutant filtration of NO_2 and O_3 (Chen *et al.* 2005). Purification technologies are also employed as a remediation strategy, these techniques include Ozonation, UV-photolysis and photocatalytic and Non-Thermal Plasma (NTP) reactions, which can remove a specific range of VOCs, but are ineffective for other air pollutants in most cases. However, these are not widely available in commercial infrastructure and can generate harmful by-products (Luengas *et al.* 2015; Irga *et al.* 2017; Pettit *et al.* 2019; Masi *et al.* 2022). NO_2 is a common pollutant released during combustion reactions as such is readily released as a vehicle emission, despite the global implementation of vehicle emission controls ambient NO_2 concentrations are seen to frequently exceed WHO concentrations of 200 µg/m3 & 40 µg/m3 (short-term: 1 h mean & long-term; annual mean) within urban centers (Hoek *et al.* 2013). Frequent exposure at these concentrations is associated with reduced pulmonary function and has been linked to increases in all-cause mortality and hospital emissions (Henschel and Chan 2013). Additionally, NO_2 acts as an O_3 precursor, oxides of nitrogen are highly reactive under high UV/temperature conditions, oxides react with methane and volatile

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organic compounds in the atmosphere to form O_3 (Lawson *et al.* 2011). Even acute exposure to O_3 can affect lung capacity, and cause reactivity in individuals with bronchoactive challenges (Lippmann 1989). Thus, it is crucial to develop technologies that can manage NO_2 and O_3 levels within indoor breathing zones, as well as near emission sources.

Plants have empirically demonstrated the ability to remove atmospheric NO_2 through both dry deposition onto plant surfaces and into the water film present on the plants' surfaces Through there stomata plants are able to directly absorb NO_2 and incorporate it into their various nitrogen pathways (Vallano and Sparks 2007), this is in conjunction with rhizospheric microbiota which can break down NO_2 into ammonia with the use of nitrogenase enzymes, this ammonia is then used to produce nitrogenous biomolecules to support plant growth (Weyens *et al.* 2015). O3, can be taken up by the plant through deposition on the cuticle. Here, O3 reacts with waxes, salt ions, and biogenic VOCs during gas phase transfer (Fares et al. 2010). However, cuticle deposition is only effective when the plant surface is highly moist; hence, stomatal absorption is regarded as the primary contributor to O3 uptake (Altimir et al. 2006; Loreto & Fares, 2007, Fares et al., 2010). The impact of this stomatal O3 absorption on plant health is not yet fully understood, but currently it is believed that upon entrance into the stomata it reacts with apoplast compounds to create reactive oxygen species (Oksanen et al., 2004).

Plants have proven effective in the phytoremediation for NO_2 and O_3 however, literature is mostly limited to studies exploring traditional urban forestry practices such as trees, hedges, and shrubs (Abhijith and Kumar 2019), and present clear variations between plant type and ability to remove NO_2. Morikawa *et al.* (1998) measured NO_2 removal of 217 different plant taxa (including several indoor plant species), the study concluded that NO_2 uptake differed as much as 657-fold between all the studied taxa identifying fireweed and *Crassocephalum crepidioides* as having the highest and third highest NO_2 content post study. While such high variations across taxa makes it difficult to reveal optimal plant characteristics for NO_2 removal, herbaceous species emerged as the more efficient plant morphotype regarding NO_2 uptake, possibly due to these plants' fast-growing nature, resulting in a greater nitrogen 'sink' then slower growing species. While only two common indoor plant species, *Spathiphyllum* sp. and *Dracaena sanderiana,* were present within Morikawa's study, they produced fairly high NO_2 uptake efficiencies. Indoor ornamental plants have been shown to effectively remove VOCs, CO_2 and PM (Tani and Hewitt 2009; Irga *et al.* 2013; Torpy *et al.* 2013; Sriprapat and Thiravetyan 2016; Hörmann *et al.* 2017, 2018, 2019, Matheson *et al.* 2023) marking them as an suitable plant species for the inclusion of novel biofilters for the removal of indoor NO_2.

Few studies have focused on the remediation capabilities of indoor plants on O_3 (Abbass *et al.* 2017), those that have attribute plant surface area and ozone deposition velocity as the primary contributing factors to ozone removal rather than characteristic that are plant species specific. In saying this, one notable species within the literature is *Dracaena deremensis* due to its high deposition velocity as well as its thick and waxy cuticles containing long-chain hydrocarbons making it a tolerant species with the ability to easily accumulate various air pollutants (Collins *et al.* 2000). However, with new forms of vegetated technology which combine plant foliage removal mechanisms and biofiltration technology in the form of 'active botanical biofilters' there is the possibility to greatly increase natural deposition velocities of any type of indoor plant, greatly increasing their efficiency to take up both NOx and O_3 from the atmosphere (Pettit *et al.* 2019).

Active botanical biofilters are one innovative green infrastructure solution currently receiving significant research. These systems use active airflow generated by fans, to pass a polluted airstream through the plant growth substrate and plant foliage (Figure 1). During this process, a proportion of the inlet pollutants are filtered by the growth media and/or degraded by the specialized microbial community living within the plant growth substrate; and may also be taken up by the plant foliage in some cases (Irga *et al.* 2018; Pettit *et al.* 2018a, 2018b). Active botanical biofilters have been shown to make functional improvements to indoor air quality environments and are an increasingly popular commercial filtration system beginning to find applications internationally and are consequently also under consideration for large scale outdoor situations (Darlington *et al.* 2001; Wang and Zhang 2011; Pettit *et al.* 2018a, 2018b). It has been conceived that active biofilters may be incorporated into conventional HVAC condition systems to increase both longevity and performance of HVAC, Typical reverse cycle air conditioning systems in buildings have an energy expenditure of USD $0.54 per hour for a medium sized 36 m^2 room. In large areas (50 m^2), HVAC systems typically cost between $0.70 and $0.95 per hour (O'Neill *et al.* 2024). A study conducted by Wang and Zhang (2011) demonstrated the potential of botanical biofilters to be integrated within HVAC systems, and while detailed cost assessments of these systems varies in relation to biofilter design several proof-of-concept studies suggesting active biofilters can contribute to HVAC energy cost savings up to 40% (Halwatura and Jayasinghe 2008; Piro *et al.* 2018; Cascone 2019; Trovato *et al.* 2020; Bevilacqua *et al.* 2021; Joshi and Teller 2021; Nadeeshani *et al.* 2021; Bruno *et al.* 2022).

Manipulative outdoor field trials of active green walls are scarce, however, research by Pettit *et al.* (2021) provided a proof of concept for the use of active green wall technology to be able to effectively filter gaseous pollutants such as NO_2, O_3 and particulate matter (PM) from road traffic emissions. However, to date, these systems have not found widespread use as an outdoor remediation system, as their purification capacity in outdoor spaces is yet to be sufficiently quantified, with single pass removal efficiencies (SPREs), especially for O_3 and PM, being insufficient to provide substantive ambient effects unless very large systems are used. Therefore, the further development of active green wall technology to increase its capacity to filter these urban pollutants is of great value for sustainable urban projects.

Rhizospheric bacteria and substrate adsorption capabilities are considered the primary sinks or pollutant removal within active botanical biofilters, with plant species selection playing an important, but less significant role in determining

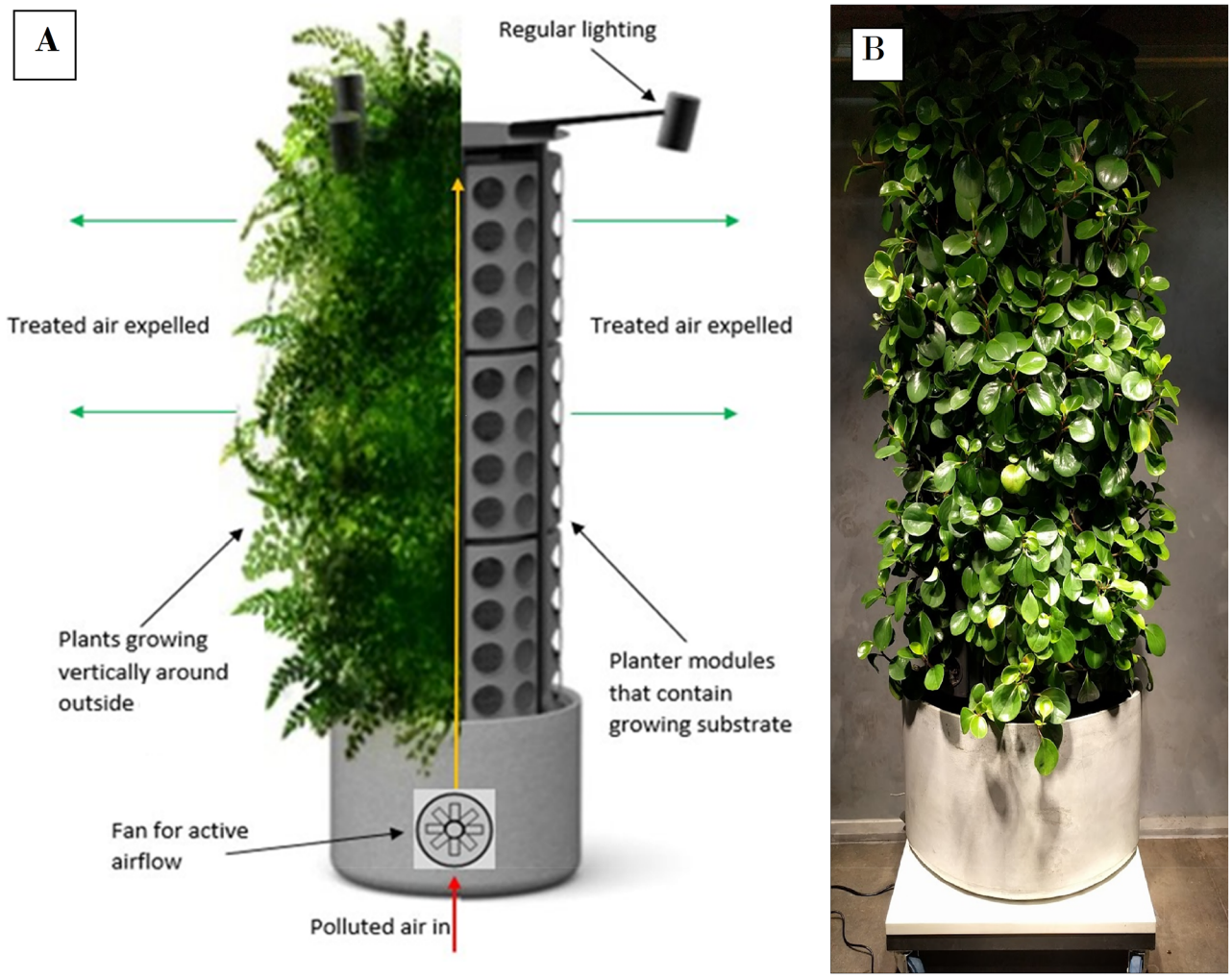

Figure 1. A) Schematic and cross-section of botanical biofilter. Air is pulled in through the base, directed up through the plenum and out through the plants and substrate and returned to ambient. B) Photo of actual system.

pollutant removal rates (Wood *et al.* 2006). Microbes within the rhizosphere are the main driving force for redox reactions which break down various N compounds into forms of ammonium (NH_4^+) and nitrate (NO_3^-) which are usable products to assist plant growth (Jetten 2008). Microbially mediated denitrification is facilitated by four groups of enzymes including nitrate reductases (narG enzymes), nitrite reductases (nirK enzymes), nitric oxide reductases (norB), and nitrous oxide reductases (nosZ) (Mohan *et al.* 2004), these enzymes are common traits of aerobic bacteria and Archaea in the rhizosphere zones, abundance and community composition of these N-type circulating functional genes is known to be influenced by the nitrogen deposition within the soil environment (Mohan *et al.* 2004), as such it is important for biofilters involved in NO_2 to effectively saturate there soil environments to produce ideal microclimates for these nitrogen degrading bacteria. Thus, substrate manipulation is considered the most effective method for improving biofilter performance. While rhizospheric degradation of O_3 has not been studied, literature has looked at the possible negative effects O_3 on plant productivity, Wang *et al.* (2024) conducted a study looking at the effects of O_3 fumigation on poplar seed rhizosphere bacterial community, interestingly they found the bacterial community shift toward enzymes for nitrogen acquisition producing more LAP and NAG to break down organic nitrogen sources (Sinsabaugh *et al.* 2008). It must be noted these communities were directly exposed to O_3 without plant mechanisms such as roots within the soil, due to the highly reactive nature of O_3 its most likely degrade into various compounds shortly after plant deposition before it can be translocated to the rhizosphere.

Previous studies have highlighted Granular Activated Carbon (GAC) as an excellent adsorbent for gaseous VOCs in botancal biofilters (Aydogan and Montoya 2011). GAC is an inert porous media with a high surface area and has shown great promise for the removal of hydrophobic VOCs in previous research (Wei *et al.* 2017). GAC can be made from any organic material containing carbon, *e.g.,*wood, corn stalk or coal, and is produced by pyrolising the material in an inert gas atmosphere (Pietrzak *et al.* 2009). The application of GAC within botanical biofilter growing media requires it to be capable of filtering air pollutants, but also simultaneously supporting long term plant health. Wang *et al.* (2012) developed a botanical biofilter substrate of shale pebbles and GAC at a ratio of 50:50 that was able to sustain plant life and maintain effective removal efficiencies for formaldehyde and

toluene for a testing period of 300 days. The incorporation of GAC within an active green wall medium may additionally significantly improve a system's ability to remove gaseous NO_2 and O_3, boosting the system's effectiveness within outdoor environments. However, research regarding GAC use in botanical biofilters has been limited to identifying its removal capabilities for VOCs and PM, thus further research is required to identify the capabilities against several target pollutants before it can be widely used in air cleaning systems.

This study aims to build upon previous indoor and laboratory research, by investigating the effectiveness of a GAC botanical biofilter growth media to remove high concentrations of gaseous NO_2 and O_3, as well as assessing the effects of changing substrate composition and moisture levels and comparing these effects to those associated with changing plant species in standard botanical biofilter medium. This was accomplished through analysis of pollutant degradation rates using scaled-down biofilter cassettes which contained various ratios of GAC incorporated into a coarse coconut husk biofilter substrate. Granular activated carbon has also been associated with PM production due to possible aerolisation of fine particles from the GAC pellets (Pettit *et al.* 2018b), thus ambient PM was also monitored as a safety protocol to observe any PM generation from the differing GAC cassettes.

Methodology

Cassette design

Scaled down experimental biofilter cassettes were used to facilitate many independent replicates and ensure the elimination of carry over effects from multiple doses of pollutants that would be unavoidable if treatments were used more than once. Cassettes were constructed from polyvinyl chloride (PVC) piping (85 mm x 85 mm, 482.1 cm^3). The substrate media was held within the cassettes by a loose weave high-density polyethylene (HDPE) membrane as is used in commercial active green walls. The 85 mm diameter and depth correspond to the airflow path within active green wall units currently in commercial use and tested in previous phytoremediation work (Pettit *et al.*, 2018a).

GAC cassettes

The trial substrate matrices were comprised of different mixtures of coarse coconut husk (particles of 8–35 mm) and granular activated carbon (GAC). The following percentages of GAC within the cassettes by mass were assessed: 0, 10, 20, 30, 40, 50 and 60% by mass, with each ratio independently replicated 6 times (Figure 2). The GAC used

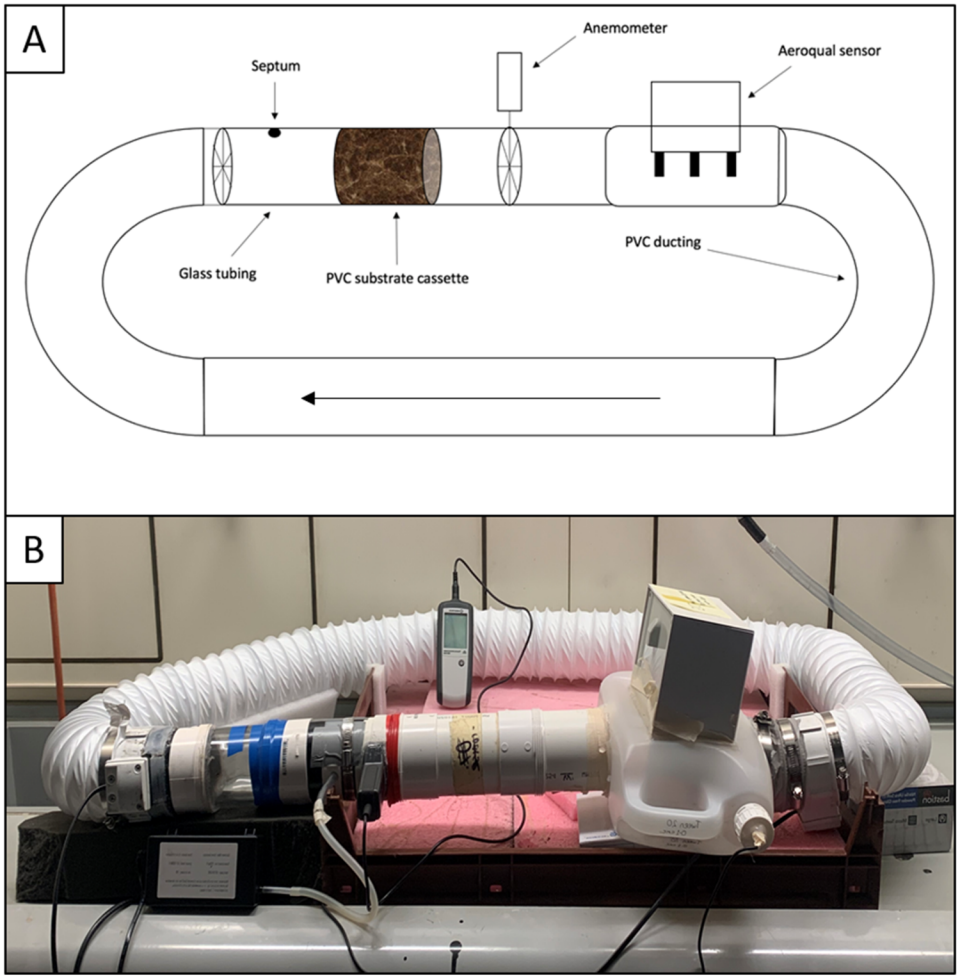

Figure 2. A) Schematic of the closed loop flow reactor used in this experiment where the arrow indicates direction of airflow, B) Photo of experimental system for the flow through testing of NO_2 and O_3.

within this work is specifically made for the removal of atmospheric VOCs and is manufactured from steam-activated coal (GAC; EA1000 4 mm; Activated Carbon Technologies Pty Ltd, Melbourne, Australia) producing its large surface area and high degree of microporosity (Activated Carbon Technologies Pty Ltd 2017). The activated carbon used had an apparent density of 0.45-0.50 g/mL, moisture as packed is 2%, surface area is 1000 m^2/g/min, and carbon tetrachloride activity is 65% min. The coarse coconut husk and GAC had bulk densities of ~ 0.20 g/cm^3 and ~ 0.52 g/cm^3 respectively.

Coconut husk has been used in several green wall studies, which found that coarse fibers were the best performing substrate for active botanical filtration systems due to their significantly lower pressure drop, enabling efficient airflow through the substrate and thus pollutant removal (Pettit *et al.* 2018a), along with a water holding capacity sufficient to maintain plant health (Paull *et al.* 2018). Coconut husk typically used within biofilters has a water content of 72.5%, is 95% organic matter, has a specific surface area of 0.75 m^2/g, and has a water holding capacity of 5.5 g [H_2O]/g dry material. All cassettes were watered to field capacity on the day of construction and then with 500 mL each morning for two weeks to remove residual fine particles that may have compromised subsequent testing. A procedural control ($n=6$) was also used which was a cassette containing no plant medium or substrate.

Plant cassette design

Experimental cassettes housing the two-plant species *Spathiphyllum wallisii* and *Syngonium podophyllum* were made using the same design as the GAC cassettes and contained a solely coarse coconut-husk substrate media packed to the same 85 mm depth of the cassettes. Each species had an averaged total leaf area of 22 cm ± 0.01, averaged root length of 22.56 ± 7.43 cm for *S. wallisi* and 8.8 ± 3.01 cm for *S. Podophyllum*. These species have been tested in similar studies (Pettit *et al.* 2019), and were chosen as they are common indoor and green wall species, with both being previously tested for their capacity to phytoremediate a range of VOCs (Tani and Hewitt 2009; Irga *et al.* 2013; Torpy *et al.* 2013; Hörmann *et al.* 2017, 2018; Matheson *et al.* 2023). General purpose 12–14-month release fertilizer was used to provide nutrient support for plant growth ([N-P-K:18-2.5–10; N as nitrate = 8.3%; N as ammonium = 9.8%; N as urea = 0%; $p=2.5\%$; K as soluble potash = 10%; $S=4\%$) with an application rate of 4 kg m^{-3} as per the manufacturer's recommendations (Australian growing solutions; Tyabb, VIC, Australia). The plants were grown in a glasshouse within their cassettes for an 8-week period prior to testing. During this period, they were stored vertically for optimal sun exposure and watered to field capacity every 4 days with an average solar exposure of 12.4 MJ m^{-2} per day and a daily photoperiod (bright sun exposure) of 7.65 h.

Closed loop flow reactor construction

For air purification testing, a closed loop flow reactor experiment was used (Figure 1a). The reactor used a sealed flow through chamber, constructed of PVC ducting, glass tubing and clear polycarbonate tubing, which has been previously used by (Pettit *et al.* 2018a, 2019). The tubular reactor had an internal diameter of 100 mm and was 2.80 m in length, with an internal volume of 0.022 m^3. Airflow was generated by two axial impellers (FANTECH TEF-100 fan, 16 W; diameter 100 mm) imbedded within the reactor, fans of similar size have been previously used in full-scale active biofilter research (Pettit *et al.* 2021). The airflow passed through the biofilter cassette and then was exposed to an air quality monitoring unit (Aeroqual AQY1, Aeroqual NZ; accurate detection limits to 0.1 ppb), which measured the average concentration of NO_2, O_3 and fine particulate matter ($PM_{2.5}$) every minute. The second axial impeller served to recirculate the air through the reactor. An anemometer (Digitech Thermo-anemometer QM1646) was fitted on the downstream side of this impeller to measure flow velocity within the bioreactor from which volumetric airflow rate was derived (Supplementary Table 1). Trials were run for 30 min to allow pollutant concentrations within the reactor to reach an asymptote across treatments, as experimental trials were of a short duration, plant and substrate characteristics outlined in the above sections remained the same after pollution exposure. All trials were conducted within a fume hood at a temperature of 22 °C and an average illuminance of 272 cd.sr.m^{-2}. After completion of the trials, waste gas was exhausted through a vacuum line in a fume hood.

Substrate moisture characteristics

Pollutant removal may also be affected by adsorbent solution interactions, as the chemical compatibility of some pollutants with water may affect their ability to adsorb onto substrate within the biofilter cassette (Liang and Chen 2010). To investigate this, two separate watering protocols were conducted before pollutant removal testing. This trial saw the GAC biofilter cassettes watered to field capacity (~ 500 mL) the night before testing, after which they were then allowed to drip dry for 24 h before experimentation. A dry trial substrate trial was performed after this, whereby GAC biofilter cassettes were air dried over two weeks within a glasshouse after which the cassettes underwent testing.

Biofiltration of high dose NO_2 and O_3

Trials for the two pollutants were run independently *i.e.*, with a single pollutant type per run, with each medium being tested for its pollutant decay rates, clean air delivery rate (CADR) and single pass removal efficiency (SPRE) of high dose O_3, NO_2 and ambient $PM_{2.5}$. To generate pollutant concentrations representative of those seen in urban areas exposed to high traffic density, high dose NO_2 was generated by placing a 1.00 cm^2 ×0.06 mm thick pure copper strip into the reactor between the fans and the biofilter cassette. After sealing the reactor, 1.50 µL of nitric acid (70% AR Grade UNIVAR, Australia) was injected through a septum onto the copper strip, generating NO_2 through the reaction outlined in Yoo *et al.* (2015). O_3 was generated by a 1000 mg

UV ozone generator (Tianchang Xingyang Electronic Technology Co, Ltd), by running tubing through a septum into the reactor. After sealing the reactor, the generator was switched on for 5 s to generate a high dose of O_3.

All biofilter cassette permutations were tested in a randomized order ($n = 6$), with additional 6 trials run without any cassettes in the reactor as a procedural control. The average peak concentrations of NO_2 within the procedural controls were 1003.0 ± 50.38 ppb, and the average peak concentration of O_3 was recorded at 1458.2 ± 51.43 ppb. These concentrations have been used in previous studies assessing non-biological methods of filtration (Yoo *et al.* 2015; Pettit *et al.* 2019).

Monitoring emitted $PM_{2.5}$ concentrations

Granular activated carbon pellets consist of compressed fine particles which have been shown to aerosolize under active air flow under some conditions (Pettit *et al.* 2018a). Reactor $PM_{2.5}$ concentrations were thus monitored during the wet and dry NO_2 trials to test the influence of different GAC compositions on the emission of $PM_{2.5}$. Average $PM_{2.5}$ ambient concentrations within the procedural control were recorded at 0.83 ± 0.23 μg.m^{-3}.

Statistical analysis

For each trial, pollutant concentrations were normalized by their peak concentrations, and exponential decay curves of pollutant concentration calculated as functions of time. The associated exponential decay rates were used as response variables for subsequent statistical analysis. One and Two-factor permutational analyses of variance (PERMANOVAs) were used to compare the decay rates of NO_2 and O_3 amongst the biofilter compositions and the trial treatments (Wet and dry) as well as the two-plant species. General linear model regressions were used to determine the influence of GAC on overall substrate water holding capacity. For each experiment, the single pass removal efficiency (SPRE) was estimated with a rearrangement of the equation in Héquet *et al.* (2017). All data analyses and graphics were performed using R version 4.0.4 (Team 2013) and the following packages: dplyr (H. Wickham 2023), ggplot2 (Wickham 2016), ggpubr (Kassambara 2023), pair-wiseAdonis (Martinez Arbizu 2020), tidyr (Wickham 2020), vegan (Oksanen 2022).

Results

GAC biofiltration of elevated NO_2

Both wet and dry trials exhibited exponential decay for the biofiltration of NO_2. Two-factor PERMANOVA revealed significant differences in the exponential decay of NO_2 amongst substrate GAC compositions ($F = 2.23$, $p = 0.04$) and moisture levels ($F = 29$, $p = 0.00$). Subsequent Tukey post hoc HSD tests showed that NO_2 decayed significantly more slowly in the procedural control compared to the 50% and 60% GAC biofilters within the wet trial (50%: $p = 0.03$; 60%: $p = 0.03$). The wetted 50% GAC composition produced the fastest decay rate for NO_2 at 0.26595 ± 0.048 s^{-1} (Figure 3), Relative decay curves are presented within Supplementary Figures 1 and 2.

The estimated CADR rates normalized by biofilter volume for NO_2 are shown in Table 1. The biofilters demonstrated the capacity to generate NO_2-cleaned air across both substrate moisture levels. There were significant differences between the two moisture levels, but not amongst the GAC concentrations ($F = 26.22$, $p = 0.00$; $F = 2.13$, $p = 0.06$, respectively). Post hoc Tukey analysis revealed that the 60% and 50% wetted cassettes produced significantly more air free of NO_2 than the control treatment ($T = 4.7$, $p = 0.02$; $T = 5.7$, $p = 0.00$) with the 50% wetted cassette having the highest CADR.

GAC biofiltration of elevated O_3

All biofilter substrate compositions demonstrated exponential decay rates for O_3 removal (Figure 4), however two-way PERMANOVA analysis revealed none of the GAC cassettes across both moisture treatments removed O_3 at significantly faster rates then the procedural control ($F = 1.77$, $p = 0.12$), indicating that effective biofiltration of this gas was not achieved. Both dry and wetted GAC decay curves are presented in Supplementary Figures 3 and 4.

Botanical biofiltration of NO_2 and O_3

Decay constants

One-factor ANOVA followed by Tukey Post hoc HSD comparing NO_2 decay rates between biofilters with the two different plant species revealed that the control had a significantly slower decay rate then the two plant treatments, *Spathiphyllum wallisii* ($p = 0.00$) and *Syngonium podophyllum* ($p = 0.00$), but no significant difference in decay between the two species was detected ($p = 0.44$; Figure 5). For O_3 decay, one-factor ANOVA indicated that neither *Spathiphyllum wallisii* or *Syngonium podophyllum* removed O_3 at significantly faster rates then the procedural control ($p = 0.59$; $p = 0.09$; Figure 6).

Clean air delivery rates

The estimated CADRs for high dose NO_2 were normalized by biofilter substrate volume and are presented in Table 2. Both plant species demonstrated the ability to produce air with reduced concentrations of NO_2, however, they did not significantly differ from each other ($p = 0.08$).

3.4. Ambient $PM_{2.5}$ monitoring

Each cassette treatment's contribution to ambient $PM_{2.5}$ are present in Supplementary Figures 5 and 6. The ambient $PM_{2.5}$ level in the control treatment was used as a zero, which was 0.83 ± 0.23 μg.m^{-3}. There was no significant difference between the wet and dry trials ($F = 1.23$, $p = 0.27$).

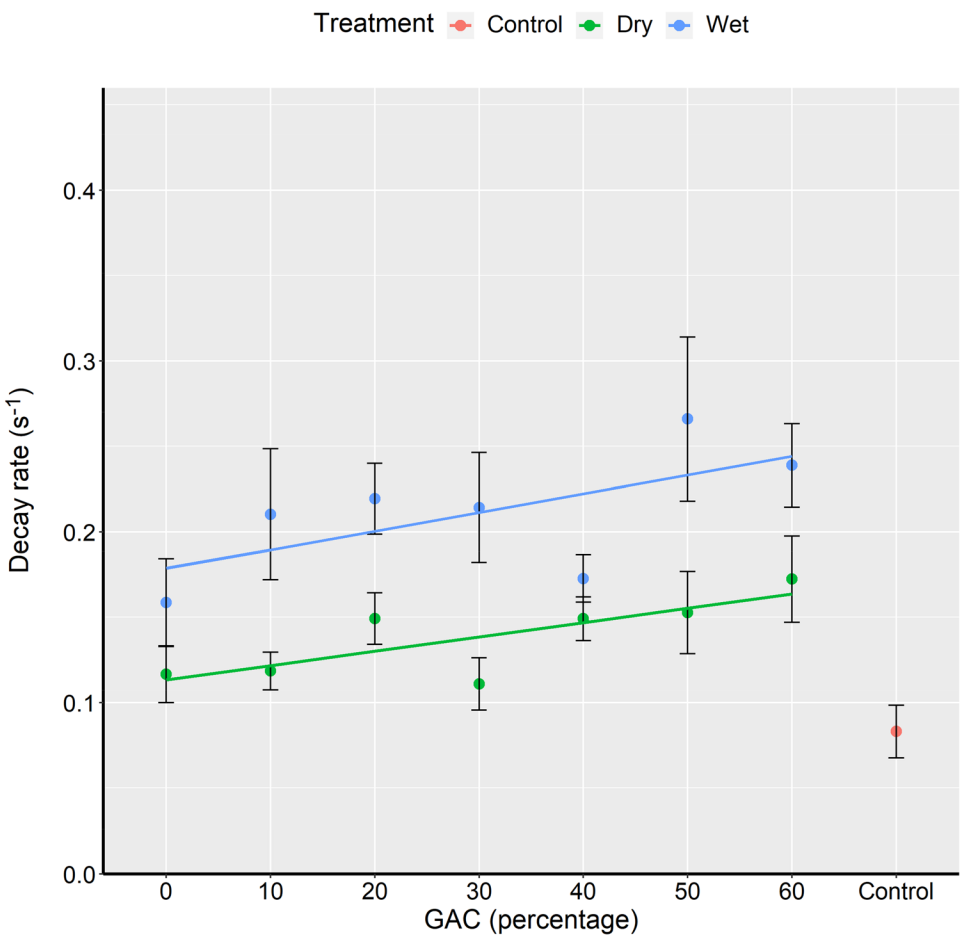

Figure 3. The average decay rate constants for NO_2 for GAC substrate cassettes across both watering treatments. $n = 6$ independent samples per treatment, error bars represent the SEM.

Table 1. Estimated CADRs normalized by biofilter volume for the NO_2 biofiltration trial ($m^3.h^{-1}.m^{-3}$ of biofilter substrate). Values are the average CADR ± SEM.

GAC Composition (%)	Dry ($m^3 \cdot h^{-1} \cdot m^{-3}$)	Wet ($m^3 \cdot h^{-1} \cdot m^{-3}$)
0	419.7 ± 59.9	576.7 ± 92.5
10	426.5 ± 39.7	762.1 ± 138.1
20	537.1 ± 54.4	823.9 ± 74.9
30	399.2 ± 55.1	755.4 ± 115.7
40	536.7 ± 46.4	577.4 ± 92.3
50	549.6 ± 86.5	1013.0 ± 173.1
60	620.4 ± 91.0	901.6 ± 87.7
Control	445.0 ± 17.0	460.4 ± 9.97

Within both the wet and dry trials, none of the treatments significantly differed from ambient levels in the laboratory ($F = 2.63$, $p = 0.51$; Supplementary Figure 1), and thus the trial systems did not contribute to $PM_{2.5}$ emissions.

Discussion

All biofilter substrate compositions across both wet and dry treatments exhibited the ability to remove NO_2 but not O_3. Within both trials, the cassettes containing higher proportions of GAC performed the best for removal NO_2. Interestingly, the influence of different watering protocols led to significant differences in NO_2 removal capacity. Substrate cassettes that underwent watering before testing removed NO_2 more rapidly than cassettes that were allowed to dry, with the 50% wetted cassette removing NO_2 most efficiently. This coincides with findings from Wang *et al.* (2012) that showed that a wetted substrate media within botanical biofilters can lead to greater removal rates of some pollutants, due to their dissolution into the moist substrate media. The differences in decay between the two moisture concentrations may be due to the solubility of NO_2. NO_2 reacts readily with H_2O, thus the higher water content of the biofilter in the wet trial could have acted as a 'wet scrubber' in removing NO_2 from the airstream (. Wang *et al.* 2012), resulting in a slightly improved degradation rates across all compositions. The addition of GAC may enhance this process, as water molecules cluster within its hydrophilic pores that would otherwise encumber is ability to adsorb hydrophobic NO_2 (Liang and Chen 2010). Consequently, the larger GAC surface area seen in the 50% composition may have provided more sites for this adsorption to take place, resulting in it providing the highest decay rate for NO_2.

Interestingly, despite there being more adsorption sites within the 60% composition, it did not outperform the 50% GAC composition medium for NO_2 removal. This may have been caused by a rate limiting step that occurs within botanical biofilters; a plateauing of NO_2 decay rates at 50% GAC has been observed in previous studies, albeit for VOC removal (Wang and Zhang 2011; Pettit *et al.* 2018b). These previous studies attributed rate limitation to the transferal of

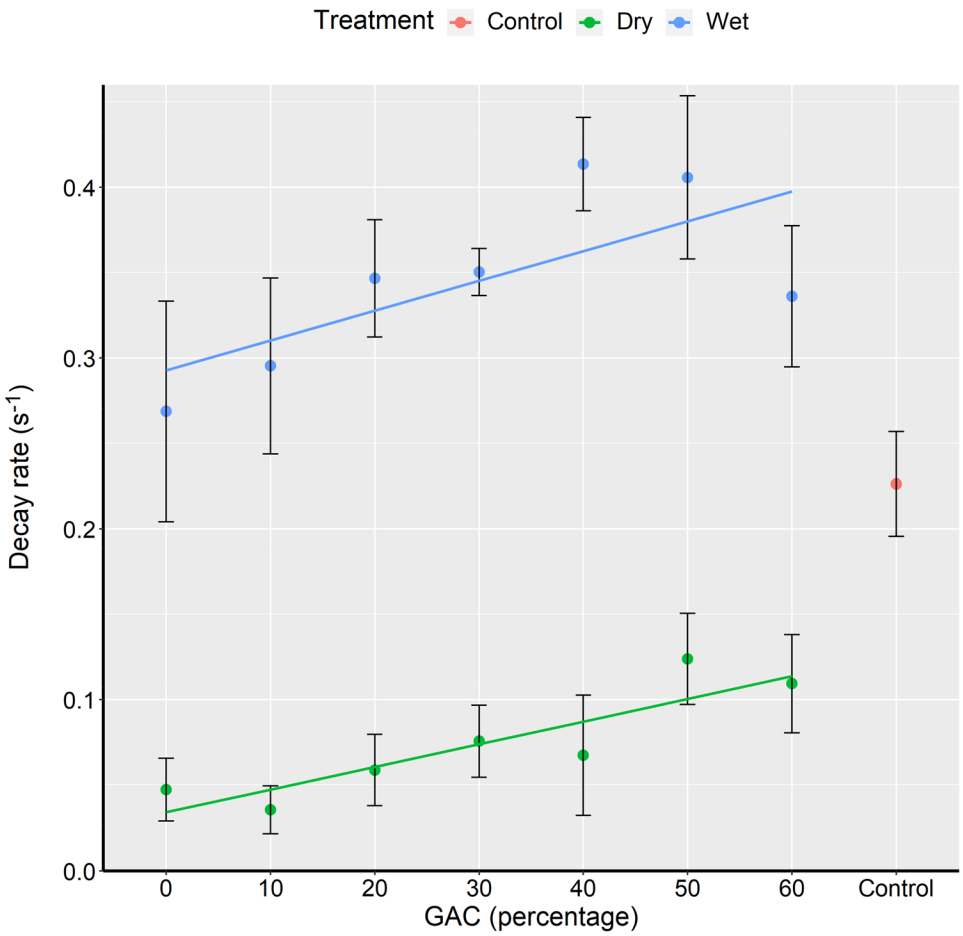

Figure 4. The average decay rate constants for O_3 for GAC substrate cassettes across both watering treatments. $n = 6$ independent samples per treatment, error bars represent the SEM.

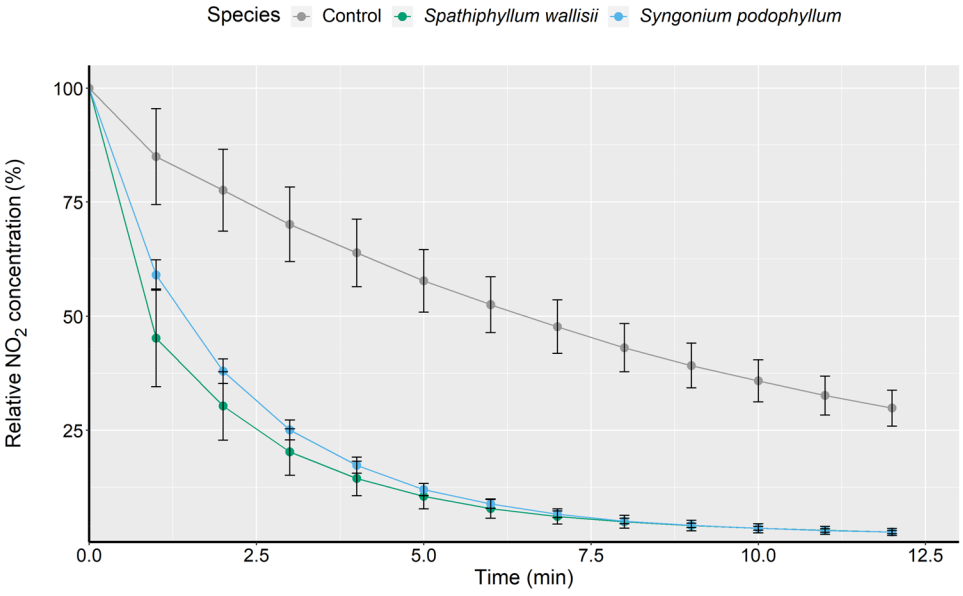

Figure 5. The biofiltration of high dose NO_2 by *Spathiphyllum wallisii* and *Syngonium podophyllum*. Error bars represent the SEM.

pollutants from the gas to the aqueous phase before they could be adsorbed onto the GAC (Halecky *et al.* 2016). Although these studies examined VOC removal which could be expected to have different absorption characteristics to the gases tested in the current work, the similarities in results suggest this limitation also occurs in the GAC NO_2 adsorption process. Importantly, for practical use, GAC is also known to be capable of removing several important

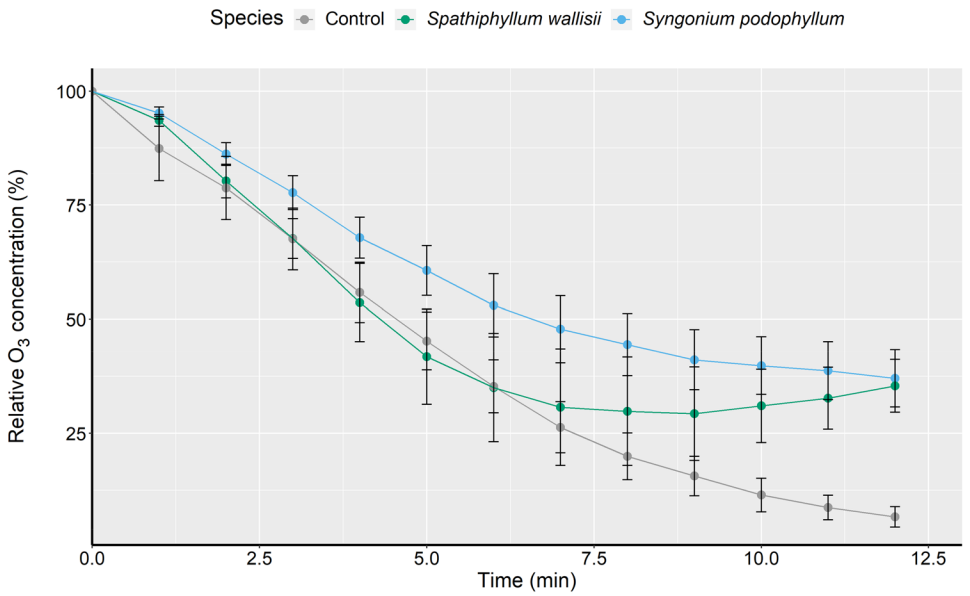

Figure 6. Biofiltration of high dose O_3 by *Spathiphyllum wallisii* and *Syngonium podophyllum*. Error bars represent the SEM.

Table 2. Estimated CADRs normalized by biofilter volume for NO_2 by biofilters containing two plant species ($m^3.h^{-1}.m^{-3}$ of biofilter substrate). Values represent averages ± SEM.

Plant species	NO_2 CADR ($m^3 \cdot h^{-1}\ m^{-3}$ of biofilter substrate)
Spathiphyllum wallisii	661.32 ± 53.28
Syngonium podophyllum	550.8 ± 19.08

plant nutrients such as ammonium, nitrates and phosphates, which could have consequences for plant health. However, Wang and Zhang (2011) tested a 50:50 mix of GAC and shale pebbles, detecting sustained plant health for over 300 days. Similarly, Pettit *et al.* (2018b) used a 50:50 mix of GAC and coarse coconut coir and recorded good plant growth for 280 days without subsequent fertilization, all while maintaining removal rates, suggesting GAC concentrations at this level are appropriate to sustain plant health and removal efficiency.

During the elevated O_3 pollution trials, no significant difference was seen between the biofilter compositions and the control ($F = 1.77$, $p = 0.12$), indicating that effective O_3 biofiltration was not occurring in this trial. This contrasts with the findings of Pettit *et al.* (2021), who recorded positive SPREs for O_3 by *in-situ* active green walls located alongside busy roadsides. However, removal rates in Pettit *et al.* (2021) differed between sites and ambient concentrations, suggesting removal may be concentration-dependent. O_3 concentrations within the current trials were elevated above normal ambient concentrations, suggesting there may be a threshold concentration of O_3 that cannot be filtered by such systems (Pettit *et al.* 2021). Additionally, it has been suggested that biogenic VOCs and NO_x can scavenge O_3, with the relationship between NO_x and O_3 being highly variable as NO_x can promote O_3 formation in the presence of light, high temperatures and gases such as VOCs. These effects make it difficult to compare studies, especially those performed *in situ* where larger air volumes would prevent VOC accumulation and NO_x concentrations would be considerably lower than within the current work. Decay of O_3 within the control may have resulted from its interaction with Ambient NO_x remaining within the flow reactor, forming species such as NO_3 and N_2O_5 (Atkinson and Carter 1984). Small amounts of gaseous O_3 were also seen to be emitted from the treatment filters within the wet trial after 4 min within the reactor, this may have been caused by a weak dissolution of O_3 in the water within the biofilter that dissociated over the course of the experiment back into gaseous O_3. Future experiments should monitor the long-term ability of O_3 to be removed within active green walls and potential of O_3 re-emission.

Both *Spathiphyllum wallisii* and *Syngonium podophyllum* biofilters exhibited higher decay rates for NO_2 than most of the purely substrate cassettes. This was possibly due to the presence of a root structure leading to reduced air-filled porosities within the substrates and thus slower airflow rates (Supplementary Table 1), resulting in higher gas-residence time and more pollution removal *via* adsorption/absorption (Pettit *et al.* 2019). Although microbial degradation of NO_2 has not explicitly been demonstrated in these systems, microbial metabolism within the rhizosphere of the root bed is considered the primary removal mechanism for VOCs and the hydrocarbons associated with PM. Due to the short duration of the current experiment, it is likely that NO_2 removal was rather driven by abiotic mechanisms such as adsorption or solubilization (Pettit *et al.* 2019) than biotic, especially metabolic, processes (Zheng *et al.* 2016). Within the observable period of this work there was no re-emissions of NO_2 suggesting possible saturation limits of the abiotic removal mechanisms was not reached, it is plausible saturation limits may be reached through the introduction of higher NO_2 concentration then were present within this work, however, the concentrations in this work are already considered elevated in comparison to average ambient concentrations. It is important to mention that substrate NOx

emissions have been detected from agricultural soils, being driven by precipitation and soil fertilization (Bertram *et al.*, 2005). However previous research on active botanical biofilters containing GAC, performed by Pettit *et al.* (2021), investigated, over the course of 6 months, the removal of NO2 by a roadside filter located within Sydney, Australia, this research period was conducted during the 'black summer' bushfires within Australia leading to elevated ambient NO2 concentrations above the yearly average. During this time the biofilter saw no-remissions of NOx, demonstrating the high pollution saturation limits of these systems when GAC is incorporated. Whilst further work is clearly required to determine the fate of NO2 in botanical biofiltration systems, proof-of-concept for removal has clearly been demonstrated, warranting this further research.

The 50% wetted GAC substrate composition produced a significantly larger NO_2 CADR ($1013.0 \pm 173.1\ m^3 \cdot h^{-1} \cdot m^{-3}$ of biofilter substrate) than biofilters containing the two-plant species, *Spathiphyllum wallisii* ($661.32 \pm 53.28\ m^3 \cdot h^{-1} \cdot m^{-3}$ of biofilter substrate) and *Syngonium podophyllum* ($550.8 \pm 19.08\ m^3 \cdot h^{-1} \cdot m^{-3}$ of biofilter substrate). CADRs provide the best estimation of the air cleaning potential of botanical biofilters. However, this experiment incorporated scaled down biofilters—larger-scale systems would clearly be capable of producing proportionately larger volumes of NO_2 cleaned air. Future work should look at combining these ratios of GAC and coconut coir within larger scale planted systems for the removal of NO_2 and O_3. Previous work by Z. Wang and Zhang (2011) calculated botanical biofilter CADRs for toluene and formaldehyde, finding variations depending on airflow rate and substrate moisture. It is thus possible that the different substrate compositions and lack of plant material in the GAC cassettes cause less obstruction and higher airflow rates leading to higher CADR values in the current trials (Darlington *et al.* 2001).

Neither plant species demonstrated the ability to remove O_3 at significantly greater levels then the procedural control. This again may be due to the high concentrations tested within this work compromising removal rates within the system. Biogenic VOCs produced by the plants within such systems have also been shown to compromise removal rates, as they may interact with ambient NO_x within the closed loop flow through reactor to scavenge O_3. However, it is difficult to estimate how this would affect *in situ* scale botanical biofilters, as the accumulation of biogenic VOCs would not be as great as it was within the small, sealed reactor volume used in this work. No significant differences in pollutant decay or CADR were recorded for both NO_2 and O_3 between the two-plant species, thus any possible plant species effects on pollutant removal appear to be masked by substrate effects. In the current work, the variability in NO_2 decay effects recorded amongst the different substrate and moisture level compositions suggests that these characteristics have a more significant effect on pollutant removal than plant species.

It has been proposed that a practical limitation of GAC in active airflow air treatment systems is its propensity to aerosolise under active airflow and emit PM, constraining its practical use within botanical biofilters (Pettit *et al.* 2018). However, within this work, despite the absence of significant differences among the treatments for the removal of ambient $PM_{2.5,}$ there was evidence within the dry substrate trial that PM can be maintained below ambient levels, particularly for substrates holding > 40% GAC. Within the wet trial, PM at concentrations above ambient levels was detected, particularly in the lower GAC ratio cassettes (10, 20, 30%), while the higher ratios produced negligible PM levels above ambient. Within this work, we cannot negate the possibility that PM recordings above ambient concentrations were a result of substrate moisture characteristics rather than the aerosolization of black carbon from GAC, as there is the possibility that aerosolised water droplets could have affected the PM recordings by the optical particle counter used in these trials (Ahlquist and Charlson 1968; Pettit *et al.* 2018b). Further research should thus be directed at identifying the characteristics of such particles emitted by biofilter systems. The findings of the current work suggest that the use of GAC in active biofilter media should be target pollutant specific, and isolated to environments where the primary concern is NO_2, and based on previous findings, VOCs.

The long-term operational efficiency of botanical biofilters to remediate NO_2 and O_3 has undergone limited testing (Pettit *et al.* 2021). NO_2 reacts with irrigation water in the substrate to form HNO_3 (Zheng *et al.* 2016). The accumulation of HNO_3 within the biofilter substrate would lower the pH and may be problematic for plant health. HNO_3 oxidation onto steam-activated GAC has been shown to degrade micropores and reduce its surface area over time, adversely affecting its ability to adsorb some inorganic species from aqueous solutions (El-Hendawy 2003). A combination of this, and the possible impairment of the microbiota in the rhizosphere could reduce botanical biofilter pollutant removal efficiency over time as its exposed to NO_2. Bacteria exposed to NO_x have demonstrated decreases in both oxidized and reduced glutathione concentrations which are critical for the maintenance of functional redox properties of many intracellular bacterial enzymes (Stern *et al.*, 2013). Longer-term experimentation with a plant-and-GAC growth medium will be required to uncover the effects of long term NO_2 exposure on plant health, microbial communities and overall long-term efficiency of pollutant removal.

Current full-scale active biofilter projects have already been installed commercially (Irga *et al.* 2018; Pettit *et al.* 2019, 2020, 2021), these installations incorporate active airflow similar to the current work. Roadside active botanical biofiltration systems have been seen to lower pollutant concentrations under standard ambient concentration, specifically, the active botanical biofilter with activated carbon used in Pettit *et al.* (2021) produced positive SPREs for both O_3 and NO_2 over a 6-month sampling period alongside a motorway in Sydney, Australia. It should be noted that Pettit *et al.* (2021) used a mixture of common ornamental plants, thus, like this work, clear differences between plant species cannot be stated. However, as the CADRs produced within this work are limited to solely substrate cassettes and two plant species, care must be taken when generalizing the current findings to large installations containing many different plant species. These results, however, provide insight into the

chemical transformations associated with the biofiltration of elevated NO_2 with a GAC media. Additionally, the capacity of GAC to improve removal rates within similar botanical filters for the removal of various VOCs (Aydogan and Montoya 2011; Wang and Zhang 2011; Pettit *et al.* 2018b) continues to highlight it as a promising addition to enhance full-scale botanical biofilters implemented in sustainable infrastructure.

Summary

This study demonstrates the capacity of biofilter substrates to effectively remove NO_2 under varying composition and moisture conditions, with the most efficient removal observed when using a wetted 50:50 ration of GAC and coarse coconut husk composition. These findings support the consensus that increased substrate moisture can enhance NO_2 dissolution with the aqueous layer acting as a 'wet scrubber', while the highly porous and enhanced surface area provided by the GAC provides increased adsorption sites resulting in significantly increased biofilter pollution removal compared to the solely coconut husk composed substrate. Importantly biofilter configurations containing *Spathiphyllum wallisii* and *Syngonium podophyllum* also facilitated significant NO_2 likely due to increased residence time within the substrate and NO_2 deposition on the leaf foliage. While this study was based on short term abiotic removal mechanisms, it is well known that microbial metabolism within the rhizosphere of the root bed is considered the primary removal mechanism for VOCs and it's appropriate to suggest these mechanisms would play a significant role in NO_2 removal as well. It is therefore recommended that future research should incorporate longer removal experiments which investigate these areas, the use of 16s rRNA gene sequencing and real time PCR analysis of rhizosphere microbial communities and associated denitrification enzymes may further elucidate the role/dynamics of various rhizospheric microbial communities in NO_2 degradation.

Conversely, O_3 removal was not observed across any tested biofilter compositions, which contrasts with previous studies that documented O_3 removal by in-situ green walls. This discrepancy may be attributed to the elevated O_3 concentrations used in this study exceeding the removal threshold of the tested substrates, this threshold may have been exacerbated but the small, scaled size of the biofilter within this work as previous botanical biofilter research has demonstrated evidence of affective O_3 removal at ambient levels *in-situ* using larger systems. Future research should investigate the long-term efficacy of biofilter systems under ambient pollutant conditions and explore potential O_3 scavenging mechanisms within larger-scale implementations, this may provide a more comprehensive understanding of how these systems perform in respect to O_3 removal and there overall multi-pollutant filtration potential.

The results also highlight key practical considerations for biofilter application in NO_2 air purification. Previous larger scale studies have provided proof of concepts for the use of active biofilters as an affective urban roadside pollution mitigation tool as well as their ability to be integrated into HVAC systems to reduce energy consumption and assist in VOC and PM removal, while this work was focussed on the smaller scale, the accurate descaled biofilter dimensions alongside significant CADRs for NO_2 cleaning presented here outline a precedent for the use of similar substrate and moisture compositions within full-scale active botanical biofilters. Future work incorporating these ideas will provide important quantitative insights into the NO_2 remediation capabilities of full-scale commercial biofilters and improve upon the efficiency of these systems within practically applied environments.

Ethical approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Authors contributions

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PI and FT supervised and aquired funding. SM and TP designed the study, SM constructed research materials, SM collected data, SM analyzed the data, SM and TP interpreted the data, SM and RF drafted the original manuscript, SM, RF, TP, PI and FT wrote and edited the final manuscript.

Disclosure statement

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Data availability statement

The data sets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

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