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Potential volatile organic compounds emission in indoor urban farming: a case study

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Abstract

A possible solution to cope with climate change and food insecurity is city greening, through extensive adoption of green infrastructures and urban agriculture. Little consideration is given to potential impacts that could be derived from elevate Biogenic Volatile Organic Compounds (BVOCs) released by plants. BVOCs can account for up to 90% of global VOC emissions. In indoor spaces, their levels are still unknown, although they may raise as much concern as for GHGs. The study presents the monitoring BVOCs emitted by a mature crop of green beans (Phaseolus vulgaris L. 'Pongo') cultivated inside an integrated rooftop greenhouse (i-RTG) in the Mediterranean area. Long-term air measurements were taken by passively sampling the atmosphere inside the i-RTG and in an inner open chamber hosting plants to monitor physiological emissions, as well as from the external outdoor environment, as control. An additional short measurement was taken on four plants in static-head space conditions to check detected BVOCs. Among a wide range of different volatiles terpenes, methanol and acetic acid were always found (including in the control), suggesting that their origin should not be associated with i-RTG plants. However, the detected signals were below the analytical procedure's LLOQ (lower limit of quantitation). In the static-head space sample, most GC-MS signals could be identified as terpenoid compounds based on their MS spectra and by comparison of their chromatographic retention times with standards; though signals were faint, estimated values were below ppb. Accordingly, the results suggested passive sampling as a practical and easy-to-implement method to produce preliminary tracking of BVOC emissions. However, active sampling may improve the quantitative assessment of their levels inside the i-RTG.

Keywords: BVOCs, i-RTG, green bean, protected agriculture, hydroponic cultivation

INTRODUCTION

Implementing urban green spaces represents one of the strategies embraced by the European programme Horizon 2020 to contain climate change impacts and improve citizens' life quality (European Commission, 2019). In this framework, urban agriculture supports city resilience and liveability (Armanda *et al.*, 2019) and simultaneously tackles issues related to food security and sustainable food production (Langemeyer *et al.*, 2021; Opitz *et al.*, 2016). Due to the limited land available for farming, innovative configurations for urban agriculture that make smart use of existing buildings and available resources have emerged in the last

years (Orsini et al., 2014). Nowadays, multiple urban farming systems are available, including indoor, open-air or mixed systems, eventually combined with the urban built environment as for the cases of integrated-rooftop greenhouses (i-RTGs) (Iaquinta and Drescher, 2015; Sabeh, 2020). One direct benefit of city greening relates to plants' sequestration of tropospheric CO₂ (Jamloki *et al.*, 2021; Saeed Meo and Karim, 2022). Currently, global CO₂ levels have increased and showed a rising future tendency (IPCC, 2021). Including urban farming can therefore improve the environmental performance of the city. However, plants are also big emitters of Biogenic Volatile Organic Compounds (BVOCs), especially terpenes (isoprenoids) that are regularly released into the environment physiologically and under external elicitation (Calfapietra et al., 2013; Niinemets et al., 2004). Some studies forecasted annual levels of BVOCs up to ten times higher than VOCs emitted by anthropogenic activity (Geron *et al.*, 1995; Guenther *et al.*, 2006). Moreover, it was highlighted a disbalancing effect towards the atmosphere (Laothawornkitkul et al., 2009; Peñuelas and Staudt, 2010) connected to BVOCs, e.g., increased residence time of air pollutants (NO_x , O_3 and CH_4) (Karl et al., 2008; Peñuelas and Staudt, 2010). Indoors, only abiogenic VOCs were extensively argued by the European Regulation (European Parliament, 2021) and supported by comprehensive guidelines (European Union, 2013; World Health Organization, 2021). Regarding biogenic ones (BVOCS), few studies have been reported on plants' emissions at the urban level (Niinemets and Peñuelas, 2008; Yang et al., 2009) and still no dedicated directions about their management have been provided. Hence, to better assess plant BVOCs levels inside innovative urban farming systems we performed a study on the measurement of crop emissions inside an i-RTG of the European Mediterranean area (Pons et al., 2015). Since multiple techniques are suggested (Heiden et al., 2015; Lundgren et al., 1994) and given the semi-indoor conditions of i-RTG, we selected the passive sampling for the collection of dynamic flows of cultivated plants (Bartual et al., 1986). Finally, obtained results were compared with those provided by a proofing measurement conducted once in static headspace (Ortega and Helmig, 2008).

MATERIALS AND METHODS

Experimental setting and investigated plants

The experiment was performed from September 13 to October 30, 2018. Crop cultivation was carried out in the South-West oriented i-RTG of ICTA-UAB institute, a building situated on the campus of the Universitat Autònoma de Barcelona (41°29'51.6"N 2°06'31.2"E). Unlike conventional greenhouses, i-RTG shares the environmental flows of the building in which it integrates and communicates with the surrounding outdoor through semi-moving façades (Sanyé-Mengual *et al.*, 2014).

Plant material

Common green bean was selected for the study, a vegetable quite spread in traditional horticulture and broadly consumed by the population of the Mediterranean area. The emission profile of this plant was retrieved from collected literature (Ballhorn *et al.*, 2008; Martins *et al.*, 2019; Quintana-Rodriguez *et al.*, 2015). About 256 seedlings of *Phaseolus vulgaris* L. 'Pongo' of ±10 cm height was equally distributed on lifted perlite bags (fig 1a) inside the cultivable area (40 m²) of the 122.8 m² i-RTG. Plants fertigation with standard nutrient solution for leafy crops dissolved in rainwater was administered via a drip system, several times during the day. Plant feeding was checked daily measuring Electric Conductivity (EC) and pH of the rainy, delivered, and leached water. Climatic conditions inside the i-RTG were tracked by temperature (T) and relative humidity (RH) sensors (CS215, Campbell Scientific) and pyranometers (L202, Hukseflux) for the radiation, distributed at different

points of the greenhouse; averages of 10 minutes data were collected by data-loggers (model CR3000 and CR1000X, Campbell Scientific Inc., USA) and used by the automated system (Siemens Building Technologies Ltd) on facades opening to modulate i-RTG's environmental conditions. Routine greenhouse and crop management, including old leaves shaving, fruit harvesting, and organic chemicals treatment, were carried out for the whole duration of plants cultivation.

Chambers' design

An open chamber for BVOCs measurements was built aside in the i-RTG (Fig. 1), assembling a steel frame (2 m width x 4.50 m length x 1.70 height x 1.95 m roof height) wrapped by low-density polyethylene (LDPE) cloth divided into two halves of 9 m³ on the long side and left open on the short one. Inside a half, two lines of 32 seedlings on perlite bags (=i-RTG crop arrangement) were placed and grown under the same regime. Air was sustained by an extractor fan (170 m³ h⁻¹ airflow) positioned on the backside to improve plant to grant climatic conditions comparable to the i-RTG. Environmental parameters were recorded by sensors arranged in the chamber, two for T, displaced at the entrance and the end of the section, one for RH, and solar radiation.

A small chamber entirely closed (0.5 m width x 1 m length x 0.6 m height) of 0.33 m^3 was constructed to sample volatile emissions in static headspace conditions on a module of four plants (=one perlite bag).



Fig. 1. Arrangement of green beans cultivation inside the open chamber and a schematic representation (left) showing sensors for T (1) and RH (2), pyranometer for radiation (3), extractor fan (5) and the diffusive canister for BVOCs collection (4); static headspace sampling in the close chamber (right).

BVOCs sampling

The sampling period began in the 3rd week of transplanting, at the stems' average height of 30–35 cm, when plants were fully developed (Ballhorn *et al.*, 2008; Li *et al.*, 2017). The collection of BVOCs was performed with passive sampling carried out twice along the season, either for one week (between Sept 26 and Oct 2), or for two weeks in a row (from Oct 2 to 22), for comparative volatiles accumulation in response to the time of exposure (Bartual *et al.*, 1986). Sampling was performed with diffusive canisters, containing preconditioned stainless-steel tubes (Carbograph 1TD 60/80, Carbosieve SIII 60/80, CAMSCO), positioned in three distinctive spots: one in the midpoint of the open chamber, one in the centre of the i-RTG and one outside, on the building's rooftop, representing the *control* sample of indoor emissions (Fig. 2). At the end of the experiment, one measurement of 5 hours (10 am to 3 pm) was taken in the small chamber with active sampling to check BVOCs emitted by enclosed plants. It was performed using a coconut-shell charcoal sorbent (Anasorb® CSC, 6 x 70 mm – 100/50 mg, SKC) mounted with a drying tube (6 x 70 mm, 250 mg, 10/60 mesh, SKC) – to prevent waterlogging in the sorbent– at a suction flow between 330-450 ml min⁻¹, generated and controlled with a pump (M&C) and a mass flow meter (E-7000 Series, Bronkhorst®) system. At the end of sampling, diffusive tubes were capped and stocked at 4°C before analysis that was led by an external laboratory (Centres Científics I Tecnològics, Universitat de Barcelona). Similarly, active sorbent briefly flushed with Nitrogen to prevent BVOCs oxidation was stored under the same condition; its analysis was carried out in situ.



Fig. 2. Allocation of diffusive canisters (sampling points) for the collection of the volatiles throughout the experiment.

Post-sampling BVOCs analysis

1. Diffusive canisters desorption and analysis.

Stainless-steel tubes were analysed at the Universitat Autònoma de Barcelona using thermal desorption (Unity Series 2-Ultra Series 2, Markes International) coupled to GC-MS system (TD-GC/MS Thermo Focus DSQII GC/MS, ThermoFisher Scientific). To point out eventual differences in instrument response, the first batch (Sep 26) was injected in (double) split mode, while the second one (Oct 2) in splitless. Oven conditions: 300 °C (maintained for 10 min) long primary desorption transported by helium gas at 55 ml min⁻¹; split injection at 11 ml min⁻¹ with initial temperature -27 °C in the cold trap (U-T15ATA-2S: TO-15/TO-17 Air Toxics trap, Markes International); 300 °C (maintained for 10 min) for secondary desorption transported by helium at 1.8 ml min⁻¹ into ZB-624 capillary column (ZebronTM, 60 m x 0.32 mm x 1.8 µm, Phenomenex). GC temperature program: initial temperature 40 °C (for 1 min), followed by a 6 °C min⁻¹ temperature ramp to a final temperature of 230 °C (maintained for 5 min). MS transfer line was set at 250 °C and ion source at 200 °C. Ms signal was achieved in MS scan mode at 30-300 amu. By screening selected ranges of m/z fragments, individuated peaks were then identified by MS spectra with the given NIST library.

2. Active sorbent extraction and analysis

Analysis of the active sorbent was performed in the ICTA laboratories following a procedure adapted from Ballhorn *et al.* (2008). The adsorbent material of the sorbent was transferred to a 4.5 mL glass vial. In the vial 50 μ L of a stock solution of 25 ng μ L⁻¹ of 1-bromodecane (1-Bromodecane 98% purity Sygma-Aldrich) internal standard and dichloromethane (DCM, \leq 100 % purity, Supelco) was added and bring to volume with DCM.

The resulting solution was analyzed using GC-MS system (7890A/7975C GC/MS, Agilent). An aliquot (1µL) was splitless injected in the GC capillary column Ultra Inert DB-5 MS (25m x 0.25 mm x 0.25µm, Agilent) carried by He at 1 mL min⁻¹ constant flow. BVOCs were eluted using the following GC temperature program: an initial temperature of 40 °C (for 2 min) followed by a 6 °C min⁻¹ temperature ramp to a final temperature of 280 °C (maintained for 4 min). MS transfer line and ion source were set at 250 °C and 150 °C, respectively. Ms signal was acquired in MS scan mode at 35-400 amu. Identification of the chromatographic peaks was performed by comparison of the MS spectra with the NIST08 database using the MassHunter software (Agilent). Concentration estimates were obtained using peak area ratios between the target compound and the internal standard, and assuming the same response factor for both compounds.

RESULTS AND DISCUSSION

Environmental conditions

Throughout the crop cycle, in the i-RTG average T and RH were 22.5 °C and 65.1 %, though peaks below and above average T and RH were registered (12.2– 39.5 °C; 18.5–94.1 %) due to seasonal climatic variations. Between the first and the second term of sampling, average T and RH progressed inversely (-3°C; +3%), according to fall progression. In the open chamber, temperature was equally distributed, and levels were equivalent to i-RTG's ones, except for relative humidity, usually higher (+6%). Average radiation registered in the i-RTG was barely 22% of the total outdoor radiation and even lower inside the chamber.

Samples analyses

1. Passive sampling results

Tentatively identification of volatiles in the two sample batches is reported in Table 1. Due to the huge number of trapped compounds and the high variability of related peak areas in the TIC (Fig. 3), BVOCs researched focussed on those reviewed by literature.



Fig. 3. Total Ion Chromatograms (TICs) of the open chamber, i-RTG and rooftop-*control* sample, obtained after one week (left) and example of extracted MS channel (right) for monoterpenes (92.50-93.50 m/z).

Quantitation of identified compounds was not possible due to signals below the quantitation limit set by the instrument method. Only preliminary information was given by the peaks' area (Table 1). Overall, no appreciable differences were detected among the batches nor between the sampling points (open chamber, i-RTG and rooftop). In the *control*

sample, a progressive increase in peaks retention times (RT) was usually observed and was attributed to tube breakthrough by particles other than VOCs (e.g., water droplets). Several peaks were identified with VOCs commonly present in the air (omitted in the summary table). Certain BVOCs were identified with monoterpenes, found in all samples with a variation of less than 40% and with α -pinene as the most abundant. Methanol and acetic acid were also ubiquitous and from 15 to 172-folds higher than terpenes but considering multiple sources emitting, their biogenicity was unconfident. Air pollutant, toluene, was present in all the collected emissions, with variable abundance, higher than most terpenes. The remaining peaks were not identified due to the faint signal-to-noise ratio (S/N) ratio.

Compound	Compound	RT	Target ion range	relative abundance range	Sampling
type	name	(min)	(m/z)	(Area counts)	spot ^a
Monoterpenes	a-pinene	19.90	92.50-93.50	390000 - 660000	A, B, C
	β-pinene ^₅	21.61	92.50-93.50	56600 - 75800	A, B
	cumene	22.46	92.50-93.50	54000 – 71600	А
	limonene	23.07	92.50-93.50	54700 - 63900	A, C, B
Benzenes	toluene	14.57	92.50-93.50	220000 – 233000	C, B, A
Aldehydes	acetic acid	11.70	59.50-60.50	5700000 - 11000000	C, B, A
Alcohols	methanol	4.62	30.50-31.50	8800000 – 10970000	A, C, B

Table 1. Identified compounds in the diffusive canisters after one week and two weeks of exposure

^aIn order of abundance, the samples obtained from A=open chamber, B=i-RTG and C=rooftop after one week of exposure. ^bLow matching percentage with the NIST library

2. Active sampling results

Relevant detected and identified compounds are resumed in Table 2. Among co-eluted signals, in the first 15 minutes of the chromatographic run, separated peaks were attributed to α -pinene, 3-carene, and limonene monoterpenes with a good matching (72-83%) with the NIST08 library. IS (1-bromodecane) was identified (>96% score) confirming the reproducibility of the analytical method performed. Detected peaks of representative mass-to-charge 100 and 173 m/z were not identified due to poor matching. (Fig. 4).



Fig. 4. TIC of the close chamber sample. In the magnification terpenes region and identified compounds are highlighted; in the chromatogram, relevant peaks comprise unknown compounds and the internal standard (IS), with target m/z ion and RT on top.

By comparison of detected peaks' area, terpenes abundance was between 4 and 53% of the IS. From the known concentration of the IS, terpenes amount was estimated to be 0.4 -

4.9 ppb (0.1 – 1.6 μ g) in the chamber. On the contrary, levels of unidentified compounds were found to be 1.0 – 11.8 ppbv (0.3 – 3.8 μ g).

Compound	Compound	RTª	Target ion	Relative abundance
type	name	(min)	(m/z)	(SNR) ^b
Monoterpenes	a-pinene	7.25	93.1	12.2
	3-carene	9.22	93.1	6.1
	limonene	9.78	57.1	26.5
Internal standard	1-bromodecane	17.57	135.1	87.2
Unknown	unknown 1	23.29	173	103.8
	unknown 2	27.84	100	27.2
	unknown 3	31.18	100	7.8
	unknown 4	43.59	173	6.2

Table 2. Detected and identified compounds in the close chamber with active sampling.

^aRetention Time.

^bSignal-to-Noise-Ratio.

CONCLUSIONS

From the study performed the highlights derived were, for the passive sampling:

- Detected BVOCs could not be attributed with certainty to the i-RTG crop, since they were found also outdoors and were not quantifiable through the method used.
- Despite occasional peaks of temperature and relative humidity inside the open chamber and in the i-RTG, which excite as elicitors on volatiles release (Niinemets *et al.*, 2004), and cumulative collection, there was no significant difference between indoor and outdoor total emissions. Especially in the i-RTG, the role of solar radiation on plant emissions is unclear or at least its impact is not demonstrated.
- The similarity found between indoor and outdoor volatile emissions suggested frequent air turnover in the i-RTG, decreasing the concentration of indoor volatiles.
- Resemblance encountered within the open chamber and the i-RTG confirmed the reproducibility of the passive sampling method for this context, though resemblance may be also due to a lack of the technical performance of the devices (e.g., water in the tubes).
- Despite the low sensitivity, passive sampling is easy-to-perform and provides a snapshot of the volatiles population of multiple environments.

And for the static headspace sampling:

- BVOCs found from this sample confirmed green bean as an emitting species.
- The preliminary quantitative information suggested plant emissions did not exceed the ppb in the i-RTG and BVOCs were lower than other volatiles.
- Since found BVOCs levels were much lower than those (≥ppm) provided by the European Report (No 29, 2013) for indoor environments, they were not believed to convey a relevant impact on human health.
- Active sampling on a static headspace system improved both qualitative and quantitative performance of BVOCs analysis but further improvements in its application could define more accurate the chemical profile of current BVOCs and their levels inside the i-RTG.

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