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Assessment of greenhouse emissions of the green bean through the static enclosure technique

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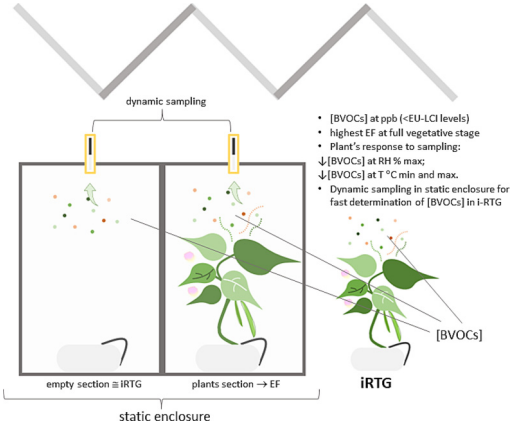
HIGHLIGHTS

- Unknown levels of indoor BVOC emissions are investigated in a building integrated rooftop greenhouse (i-RTG).
- Dynamic sampling in static enclosure is proposed for fast BVOC emissions determination in indoor systems.
- Higher BVOC emissions are expected along full vegetative development.
- Enclosing conditions may affect true emissions estimation.
- Indicative BVOC levels provided are below the lowest concentrations of interest reported in official tables.

GRAPHICAL ABSTRACT



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ABSTRACT

Urban green installations are extensively promoted to increase sustainable and accessible food production and simultaneously improve the environmental performance and liveability of city buildings. In addition to the multiple benefits of plant retrofitting, these installations may lead to a consistent increase in biogenic volatile organic compounds (BVOCs) in the urban environment, especially indoors. Accordingly, health concerns could limit the implementation of building-integrated agriculture. In a building-integrated rooftop greenhouse (i-RTG), throughout the whole hydroponic cycle, green bean emissions were dynamically collected in a static enclosure. Four representative BVOCs, α-pinene (monoterpene), β-caryophyllene (sesquiterpene), linalool (oxygenated monoterpene) and cis-3-hexenol (LOX derivate), were investigated in the samples collected from two equivalent sections of a static enclosure, one empty and one occupied by the i-RTG plants, to estimate the volatile emission factor (EF). Throughout the season, extremely variable BVOC levels between 0.04 and 5.36 ppb were found with occasional but not significant ($P > 0.05$) variations between the two sections. The highest emission rates were observed during plant vegetative development, with EFs equivalent to 78.97, 75.85 and 51.34 ng g^{-1} h^{-1} for cis-3-hexenol, α-pinene, and linalool, respectively; at plant maturity, all volatiles were either close to the LLOQ (lowest limit of quantitation) or not detected. Consistent with previous studies significant relationships ($r \geq 0.92$; $P < 0.05$) were individuated within volatiles and temperature and relative humidity of the sections. However, correlations were all negative and were mainly attributed to the relevant

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effect of the enclosure on the final sampling conditions. Overall, levels found were at least 15 folds lower than the given Risk and LCI values of the EU-LCI protocol for indoor environments, suggesting low BVOC exposure in the i-RTG. Statistical outcomes demonstrated the applicability of the static enclosure technique for fast BVOC emissions survey inside green retrofitted spaces. However, providing high sampling performance over entire BVOCs collection is recommended to reduce sampling error and incorrect estimation of the emissions.

1. Introduction

Despite the current climatic emergency and COVID-19 pandemic, the global population increase is expected to reach 8 billion by the end of 2022 and approximately 9.7 billion in the next thirty years (United Nations DESA, 2022). The consequent broadening of urbanized areas in response to an increasing number of inhabitants is driving the agricultural sector toward innovative and sustainable techniques for food production that prioritizes proximity to the city (Maye, 2019; Buscaroli et al., 2021; Rao et al., 2022). Urban planning has also moved in the direction of a more sustainable design by integrating vegetation into buildings (Appolloni et al., 2021; Puppim de Oliveira et al., 2022), both for cultivation and ornamental purposes, such as indoor farming (Sabeh, 2020; Avgoustaki and Xydis, 2021; Banerjee et al., 2022) and vertical gardening (Orsini et al., 2020; HUI et al., 2022).

Green installations have been reviewed for their positive effects on building performance in terms of temperature and noise mitigation (Li et al., 2022; Oquendo-Di Cosola et al., 2022) and energy inputs (Muñoz-Liesa et al., 2021; Zambrano et al., 2021) and considered for their capability to remove harmful volatile organic compounds (VOCs) from indoor spaces (Ysebaert et al., 2021) and decrease environmental carbon emissions (Lampinen et al., 2022; Saeed Meo and Karim, 2022). However, many studies on air assessment in the open field have reported the release of high biogenic volatile organic compounds (BVOCs) from plants (Laothawornkitkul et al., 2009; Calfapietra et al., 2013), mainly isoprene-based compounds, and their chemical interaction with the tropospheric layer. Multiple chain reactions occurring between BVOCs and air molecules may result in significant increases in atmospheric GHGs (CO_2, N_2O, NOx, O_3, CH_4) and suspended organic aerosols (Cai et al., 2021; Mahilang et al., 2021; Dodman et al., 2022), reflecting air quality and climatic condition variations (Fu et al., 2010; Peñuelas and Staudt, 2010; Glotfelty et al., 2016).

Individuating large-scale effects dependent on plant emissions is demanding, although it could be estimated via extended monitoring and real-time sampling of the urban and wild environment (for instance PTR-MS), or via prediction models (Guenther et al., 2006), which could show particular trend distributions of biogenic fluxes (Chuang et al., 2011; Seco et al., 2015; Drewer et al., 2018; Coggon et al., 2021). In the city area the impact of BVOC emissions regarding green-integrated infrastructures was already theorized (Niinemets and Peñuelas, 2008; Tiwari et al., 2019; Persiani, 2021), but currently research targeting the assessment of BVOC levels in urban environments is limited (Yang et al., 2009; Zhang et al., 2020).

Indoors, safety levels and ventilation guidelines for anthropogenic and biogenic VOCs have been comprehensively wrapped up in harmonization frameworks of the ECA-IAQ (European Union, 2020; European Union, 2021). Based on recognized health procedures and according to collected monographs, report n°29 provides the lowest concentration of interest (LCI) for a VOC that does not reproduce an adverse effect on human health after long-term exposure in a closed environment. With respect to BVOCs, LCIs are fixed between 1400 and 5000 $\mu g\ m^{-3}$ but individual levels were determined only for few monoterpenes (α-pinene, β-pinene, 3-carene and limonene) despite broad range of volatiles emitted by plants. With respect to the assessment strategies, different sampling techniques and important elements to consider have been discussed (European Union, 1994), supporting user's choice of the most adequate sampling procedure. Since indoor BVOCs can accumulate, levels may exceed safety risk values and result in serious health issues related to breathing and hypersensitivity (Müller et al., 2002; Yang et al., 2009; Maffei et al., 2011; European Union, 2013). Generating data about their potential environmental fluxes can therefore be of great interest.

In view of prospecting scenarios and current research gaps, we carried out a quali-quantitative assessment of the BVOC emissions generated by crop cultivation in an integrated rooftop greenhouse (i-RTG) that was placed in the ICTA-UAB building (Sanjuan-Delmás et al., 2018). Following proposed strategies and previous works (European Union, 1994; Soria et al., 2015; Li et al., 2019; Tholl et al., 2021) BVOCs were collected in a static enclosure with dynamic sampling technique and recovered with solvent extraction, identified, and quantified by gas chromatography coupled mass spectrometry (GC–MS). Detected levels were compared with available LCIs as main referencing values for indoor emissions and additionally BVOCs emission factor (EF) related to the different phenological stage was calculated throughout plants development. Finally, the performance and the reproducibility of the overall sampling methodology performed was evaluated.

2. Materials and methods

2.1. I-RTG arrangement and plant cultivation

Green bean *Phaseolous vulgaris* L. 'Pongo' was cultivated in the southwest-facing greenhouse located inside the i-RTG of the ICTA-UAB research institute (41°29′51.6″ N 2°06′31.2″ E, Fig. 1). Both i-RTG and building specific configuration allow a constant exchange of environmental flows among the spaces and, alternately, with outdoor flows according to the programming of the façade system (Muñoz-Liesa et al., 2021) (Fig. 1). Crop cultivation was carried out in spring 2020 (February 12 to May 15).

The cultivation system was run hydroponically under drip fertigation and organized into 8 trailed lines of flanked perlite bags. A total of 256 seedlings of 10–15 cm height were transplanted with a plant spacing of 0.20 m × 0.40 m, resulting in a density of 15 plants per m^2 (Fig. 2). The cultivated area was approximately 40 m^2 (Fig. 2). Plants were grown with a nutrient solution for leafy crops dissolved in rainwater. Water management was controlled by a programmed irrigation system that was periodically adjusted for the plants' daily requirements. Daily water pH and electric conductivity (EC) were measured in the delivered and leached solutions and in the collected rainwater to maintain an adequate nutritional supply. Ambient temperature, relative humidity, and radiation were constantly monitored (systems CS215, Campbell Scientific and L202, Hukseflux) by averaging 10-min time periods using dataloggers (models CR3000 and CR1000X, Campbell Scientific Inc., USA). Indoor cultivar requirements were fulfilled via the movement of the façade, handled by an automated station (Siemens Building Technologies Ltd).

2.2. Static enclosure design and setup

Plant emissions were collected in a chamber, which was a static enclosure (Tholl et al., 2006) built alongside the crop inside the i-RTG (Fig. 3). Chamber dimensions were established following those of a previous test designed for preliminary emissions assessment (Stringari et al., 2022). The chamber consisted of a rectangular steel frame assembled and delimited with low-density polyethylene (LDPE) cloth. The chamber was divided into two equal sections of 1000 L (0.35 m width × 0.85 m height × 3.50 m length). The chamber remained open to the i-RTG ambient and was isolated during the BVOC sampling process. In the right section, 12 seedlings were

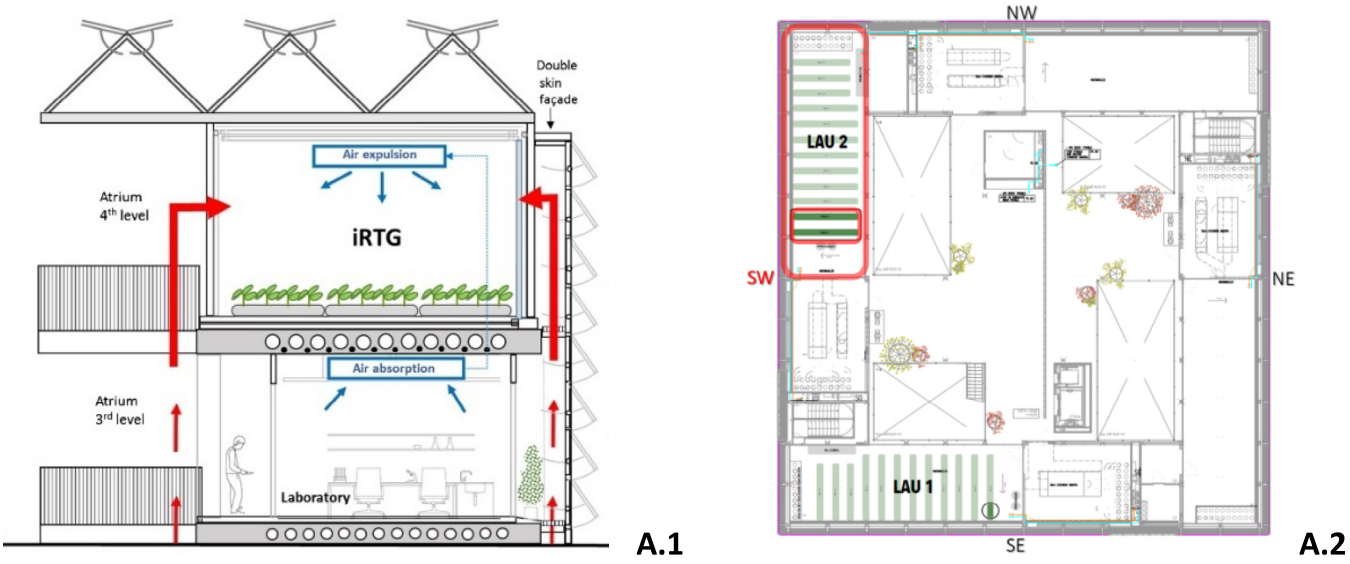


Fig. 1. Schematic reproduction of the environmental flow direction along the ICTA building, adapted from (Nadal et al., 2017). (A.1) and a planimetry of the 4th floor dedicated to urban agriculture, where current/the considered i-RTG is highlighted (A.2).

transplanted into perlite bags distributed inside the section and grown under the same environmental conditions as the i-RTG crop. The volume occupied by the single plant (0.0867 m^3) was further applied in the calculation of the EF of the detected volatiles. The left section remained empty to provide a representative sample of i-RTG ambient. Temperature, relative humidity, and radiation inside the chamber were continuously recorded to obtain supporting information regarding plant physiological status before and during the BVOC sampling process. In the empty section, only temperature was monitored.

2.3. BVOC dynamic sampling

Air BVOCs were extracted into charcoal cartridges (Anasorb® CSC, 6 × 70 mm – 100/50 mg, SKC) using a controlled air stream (Electro A.D. pump and flow meter E-7000 Series, Bronkhorst®). Drying tubes (6 × 70 mm, 250 mg, 10/60 mesh, SKC) were placed upstream of the cartridges to ensure adequate BVOC extraction. Air was collected from the center of the chamber to represent its atmospheric composition. Since our purpose was the collection of the current environmental air stream in the i-RTG, no air purging was performed inside the enclosure prior to sampling. For each sampling process, a total volume of 3.75 L of air was extracted during a 15 min time period (airflow 0.25 L min^{-1}). This term was decided after observing the environmental performance several times in the plant section, considering the well-known influence of abiotic stress on the plant emission response (Ortega and Helmig, 2008), and focusing mainly on relative humidity, which usually showed the highest variation under extended closure. The sampling period started 5 weeks after transplanting, ensuring that the average stem size was above 30 cm (March 26 until May 8). Air samples were taken multiple times during individual phenological stages of the crop cycle (vegetative-reproductive-productive-senescent), individuated by periodical stem sizing, blossom counting and biomass (fallen leaves and harvested fruits) weighing. Consecutive measurements were considered repetitions of a determined stage. Samples were obtained at or near the 12 AM time point, coinciding with the maximum plant activity (Loreto et al., 2006; Ortega et al., 2008). Additional air samples were obtained before the start of the experiment to control BVOCs in the i-RTG ambient environment. Procedural blank samples were also analyzed to identify possible contamination during the analytical process.

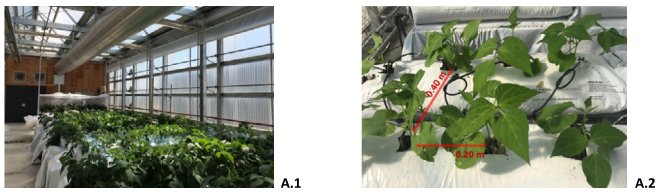


Fig. 2. Lateral view of the arrangement of the i-RTG cultivation (A.1) and a picture of the plant spacing along the substrate bags (A.2).

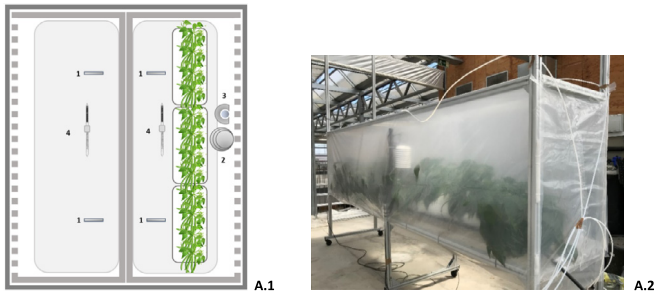


Fig. 3. (A.1) Graphical setup of the chamber for volatile collection in the empty (i-RTG) and filled (12 plants) sections (sensors 1, 2 and 3 for temperature, relative humidity, and radiation; 4 is the BVOC sorbent + dryer tube); (A.2) Picture showing the air sampling.

2.4. Post-sampling analysis

2.4.1. BVOC determination

The collected sorbents were flushed with nitrogen to prevent BVOC oxidation and then capped and stored at 4 °C before analysis.

BVOC characterization focused on four of the compounds usually emitted by green beans (Table 1) and leafy plants (Holzke et al., 2006; Fu et al., 2010; Maffei, 2010; Malik et al., 2018) and representative of their chemical class. Qualitative and quantitative analysis was performed on monoterpene α-pinene and oxygenated monoterpene linalool, on sesquiterpene β-caryophyllene and on lipoxygenase derivate cis-3-hexenol following the NIOSH Method n. 1552 (NIOSH, 1996). Then, BVOCs were desorbed with 1 mL of carbon disulfide (CS2 for spectroscopy, ≥ 99.9 %, ACROS ORGANIC) and transferred to 1.5 mL vials.

Sample extracts and standard solutions were analyzed using a GC–MS system (TRACE 1300/1310 GC coupled to an ISQ7000 MS, Thermo Scientific, USA). Extracts (2 μL) were injected in split-less mode (1 min, 240 °C), and BVOCs were separated using a TG-WAXMS capillary column

Table 1
List of typical (bold text) and induced (*) BVOCs emitted by *Phaseolus vulgaris* L. retrieved from the literature (Croft et al., 1993; Ballhorn et al., 2008; Souza et al., 2013; Quintana-Rodriguez et al., 2015).

BVOCs class				
Terpenoids		LOX derivates (lypoxygenase)	Shikimic acid derivates	Photosynthesis derivates (glucose)
Monoterpenes	**α-Pinene**	**cis-3-Hexenol***	**Methyl salicylate***	**Methanol***
	β-Pinene*	**cis-3-Hexenal***	**Indole**	
	cis-β-Ocimene*	**trans-2-Hexenal***		
	Limonene*	**trans-2-Hexenol***		
	1,8-Cineole*	trans-3-Hexenal*		
	Camphene	trans-3-Hexenol*		
	Linalool*	(1-Hexanol)*		
		Acetate (acetic acid)*		
Sesquiterpenes	β-Caryophyllene	cis-3-Hexenyl		
	L-carveol*	**Acetate***		
	cis-α-Bergamotene*	1-Octen-3-ol*		
	α-Terpineol*	2-Ethylhexan-1-ol*		
	Farnesene*	Butyrate*		
		cis-3-Hexenyl		
Homoterpenes	trans-4,8-Dimethyl-	Isovalerate*		
	1,3,7-Nonatriene*	cis-Jasmone*		
		Methyl jasmonate		
Others	**2-Butanone***			
	Nonanal*			
	Decanal*			

(TraceGOLD™, 30 m × 0.32 mm × 1 μm, Thermo Scientific, USA) with a constant flow of 2.7 mL min^{-1} of He as carrier gas. Compounds were eluted using the following temperature gradient: initial temperature of 35 °C (for 1 min), followed by 10 °C min^{-1} temperature ramp to a final temperature of 215 °C (held for 1 min). MS transfer line and ion source temperatures were set at 240 °C and 200 °C, respectively. Compounds were ionized at 70 eV and the resulting ions were detected in scan mode at 50–600 amu (0.2 scan s^{-1}) for identification purposes. Compounds were identified by comparing their MS spectra with the NIST library (NIST 17 Mass Spectral Library Software) using Chromeleon Studio 7.2 software (Thermo Scientific). Quantitation was led in timed selected ion monitoring (SIM) to improve instrument sensitivity and specificity. In SIM mode, compounds were identified by comparing the retention time with solutions containing commercially available standard compounds. The lowest limit of quantitation (LLOQ) of target BVOCs was assessed by injecting six replicates of the smallest standard solution (0.001 ng μL^{-1}) detected (LOD), with a relative standard deviation (RSD%) ≤ 21 %. (Table 1 in the Supplementary materials). BVOCs concentration (ng μL^{-1}) in the samples was calculated using a calibration curve obtained with standards (0.001–0.1 ng μL^{-1}).

2.4.2. BVOC emission factor calculation

Quantitative data obtained for the detected BVOCs were then applied to the calculation of an EF, following (Li et al., 2019) Eq. (1) for a static enclosure system: the EF (ng g^{-1} h^{-1}) of a BVOC species (EF_{BVOC}) is given by the difference between the total BVOC concentration (C, ng L^{-1}) emitted by the plant and its concentration in the background air sample (C_0) collected in the static enclosure (V and V_0, L) for the total time of enclosure (Δt), related either to the plant (P) leaf area (m^2), leaf area index (LAI), or fresh/dry biomass (g). In our experiment, no background air sample was provided prior to sampling (no air purging) inside the enclosure. Therefore, the total BVOC concentration was correct on the blank samples collected. The derived equation was then simplified (Eq. (2)), giving the EF_{BVOC} for the green bean plant.

$$EF_{BVOC} = \frac{C \times V – C_0 \times V_0}{\Delta t \times P} \quad (1)$$

$$EF_{BVOC} = \frac{C \times V}{\Delta t \times P} \quad (2)$$

The leaf area and biomass of the plants were measured periodically along progressive growth stages. Specifically, four randomly chosen plants were cut at the stem base; the whole plant, stem, and leaves were weighed to provide the average weight of fresh biomass. The 25 % of harvested leaves was gently spread onto a scan surface, and the acquired image was processed using an encoding Python 3.8 script (Spyder 4.1.5; full code in the Supplementary materials) that calculated the area of the leaves; the 100 % area was derived by relating the area to the corresponding biomass weight. The dry weight was also provided by drying the fresh biomass in a static oven (60–70 °C). The LAI calculation was derived by the ratio between the average leaf area calculated and the plant spacing in the i-RTG (0.20 m × 0.40 m).

2.4.3. Statistical analyses and environmental correlation

The reproducibility of the carried-out sampling technique was examined using statistical analyses to ensure the reliability of the results. Between the two sections of the static enclosure, BVOC emissions were compared with paired tests (Wilcoxon signed-rank test). Similarly, environmental conditions – temperature and relative humidity – monitored during the closure time were compared with one-way test (ANOVA). In addition, the effect of the static enclosure was investigated by looking at significant Pearson correlations (*r*) between environmental variables (radiation, temperature, and relative humidity) and individual BVOCs. Data processing was realized in R Studio (version 3.6.3).

3. Results

3.1. Environmental performance inside the i-RTG during plant cultivation

Far from the sampling, the mean temperature registered over the crop cycle in the i-RTG and in the empty section of the static enclosure was equivalent (18 °C and 19.5 °C, respectively) and slightly higher in the plant section (20 °C); this was also found between the mean relative humidity in the i-RTG (56 %) and in the plant section (54 %). The mean radiation measured at the crop level in the i-RTG and in the chamber was also similar (51.83 W m^{-2} and 47.90 W $m^{-2,}$ respectively). During air measurements, chamber temperature was higher in the plant section and on average 2.5 °C higher than in i-RTG; in contrast, relative humidity in the i-RTG was still higher than levels recorded in the plant section (+16 %), although these levels as well as for temperature ones were found to increase exponentially along the time of enclosure (Fig. 4) with peaks close to the saturation point.

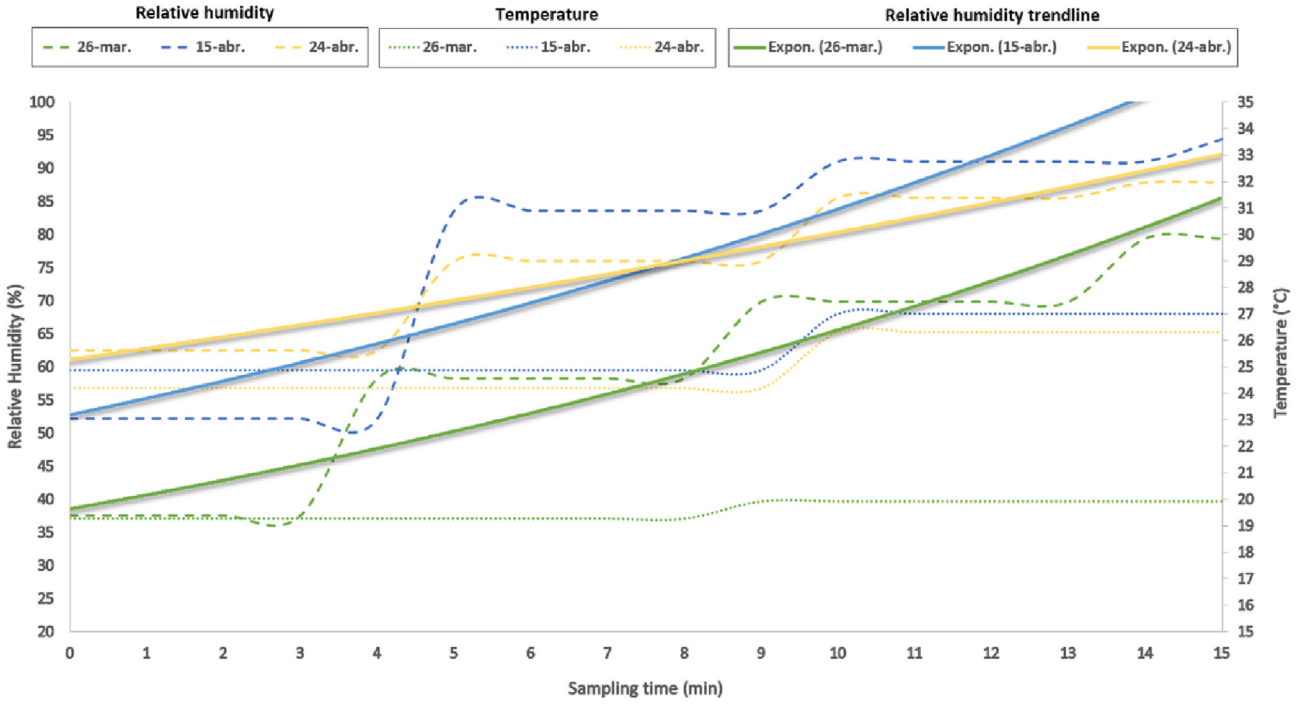


Fig. 4. Three representative records of temperature and relative humidity measured in the plant section during air sampling; exponential trendlines of relative humidity are highlighted.

3.2. Crop emission levels in the i-RTG

Throughout the cultivation cycle, the chemical profile of the collected emissions was extremely variable, ranging between 0.35 μg m^{-3} and 29.87 μg m^{-3} (0.04–5.36 ppb) or not detected (below the LLOQ). The highest concentration of each detected BVOC was compared with the respective EU-LCI value (Table 2), which was defined only for α-pinene but not for the rest of the compounds. Therefore, these levels were compared to the given values of the corresponding terpene class, except for the missing values for cis-3-hexenol. All the investigated compounds were below the most updated EU-LCI values. The collected emissions throughout the season are shown in Fig. 5. Considering BVOC abundance, monoterpene α-pinene was the most abundant (1.33–29.87 μg m^{-3}), followed by LOX derivate cis-3-hexenol (2.13–20.64 μg m^{-3}), oxygenated monoterpene linalool (0.40–16.69 μg m^{-3}) and sesquiterpene β-caryophyllene (0.35–0.61 μg m^{-3}). Considering the BVOC distribution, cis-3-hexenol and linalool were typically detected throughout the season and in both sections of the chamber; in contrast, α-pinene and β-caryophyllene were occasionally detected and more often in the empty section. Emissions differences between the two sections were highlighted by subtracting the BVOC amount of the empty section from the section enclosing the plants (Fig. 6). Although remarkable negative and positive emission variations were observed for all the detected compounds, statistical analysis did not show a significant difference between the collected emissions (lowest P value >0.36). Likewise, the environmental factors measured during collections were generally similar except for relative humidity, whose maximum and minimum values registered in the two sections were significantly different ($P < 0.001$ and $P < 0.01$, respectively). The interpretation of such variations is addressed by the environmental load analysis in the following section (*Section 3.3*).

Table 2
Compiled EU-LCI values, highest concentrations collected in the static enclosure over the crop cycle, and the instrument lowest limit of detection (LLOQ).

Compound	μg m^{-3}		
	EU-LCI value	Detected level	LLOQ
α-Pinene	2500	29.87	0.489
Linalool	1400[a]	16.69	0.391
β-Caryophyllene	1400[a]	0.61	0.320
cis-3-Hexenol	–	20.64	0.449

[a] This group includes all monoterpenes, sesquiterpenes and their oxygen-containing derivatives.

3.3. Environmental load analysis of BVOC emissions

By computing individual BVOC emissions with the mean, minimum and maximum rates of the variables recorded (temperature, relative humidity, and radiation) in compiled matrices, significant correlations were detected (Fig. 7). In both the empty and the plant sections, analogous negative correlations were found between LOX derivative cis-3-hexenol and temperature variation ($P < 0.05$); for this volatile, the same correlation was observed with maximum relative humidity rates, although only in the plant section, as well as oxygenated monoterpene and sesquiterpene, linalool and β-caryophyllene ($P < 0.01$). In contrast, no correlations were found for the monoterpene α-pinene. No relevant relation was underlined with radiation. As a result of the computational matrix, a strong positive correlation among all the volatiles ($P < 0.001$) was also highlighted in both sections. Among the variables, significant negative correlations ($P < 0.01$) between relative humidity maxima and the different patterns of temperature were individuated but only in the plant section.

3.4. Emission factor of detected BVOCs throughout the crop cycle in the i-RTG

From the emissions collected in the plant section, the respective BVOC EFs were determined and related to the corresponding crop growing stage according to phenological measurements (Table 3). General tendencies were marked, although exceptional levels were also observed. Emission bursts were associated with vegetative development completion, decreasing progressively during blossoming and fruit production until senescence, which was characterized by the lowest EFs registered.

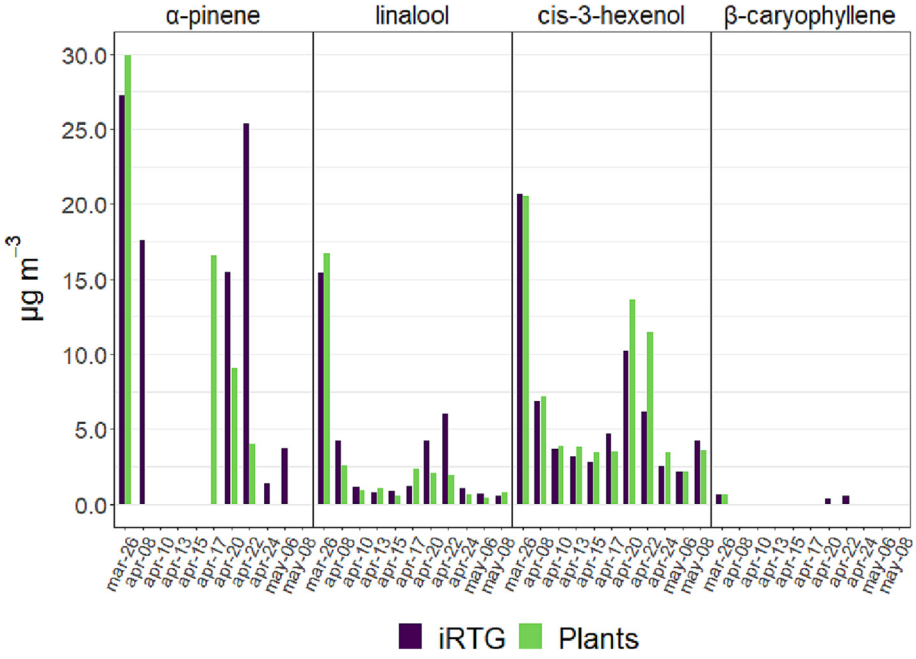


Fig. 5. Total BVOC emissions (µg m^{-3}) collected in the empty and plant sections of the chamber throughout the cultivation of the green beans in the i-RTG.

4. Discussion

Throughout the cultivation cycle, all the green bean BVOCs selected were detected. In general, levels described by measured concentrations were found to be variable. Because the building façade's screening mechanism altered the i-RTG radiation, the ozone impact on the total BVOCs collected was neglected. Concentrations of all investigated volatiles were much below ascribable EU-LCI values. Additionally, taking as a reference the highest BVOC detected (29.87 µg m^{-3} or 0.0054 ppm), α-pinene, its concentration was approximatively 15 folds lower than the Risk Value (450 µg m^{-3} or 0.0803 ppm) calculated from long exposure studies (from 6 h to 5 days). Indeed, even when researchers were in the i-RTG for long periods of time (e.g., during harvesting or greenhouse maintenance), their exposure to emissions was mitigated by frequent ventilation via automated windows and the edifice structure itself, which can contribute to a fast emissions decrease. Nevertheless, the main phenological EFs derived underlined high emissions at vegetative development and variable or low emissions over the progression of plant growth (Table 3). Individual BVOC chemistry and environmental relationships, as well as plant activity, should thus be revised. For monoterpenes (α-pinene) and sesquiterpenes (β-caryophyllene), release (Loreto et al., 2006) is typically driven by temperature and characterized by high reactive bursts (Guenther et al., 2006; Materić et al., 2015), although release is much slower for sesquiterpenes (Duhl et al., 2008), which may lead to underestimations during sampling. Emission of oxygenated monoterpenes (linalool) and LOX derivatives (cis-3-hexenol) is also generally inductive, regulated by both biotic and abiotic factors (Niinemets et al., 2013; Jamloki et al., 2021), but rather stable and more persistent in the atmosphere than hydrocarbon terpenes. In the literature, monoterpenes have been reported along the vegetative stage (Holzke et al., 2006) to progressively decrease at plant maturity (Mozaffar et al., 2018), while OMT and LOX compounds have been documented over the entire productive phase (Manco et al., 2021), which is consistent with the emission rates measured. Frequent variations were tentatively explained by the environmental load analysis in the two sections (*Section 3.3*). Considering plant section, general increase of temperature and relative humidity during the sampling could be reasonably associated to plants physiological response (transpiration) to the enclosing (Heiden et al., 2015; Pérez-Priego et al., 2015; Soria et al., 2015) and were significantly related with detected emissions. However, negative correlations found between many of the volatiles and relative humidity, and in one case (cis-3-hexenol) with temperature are in contrast with past findings and may be questionable. A possible explanation may be found in scrutinized analysis of the molecular pattern of VOCs in conditions of high humidity levels (Zhou et al., 2017). Though the combination of increased temperature and relative humidity may improve emission amount in the environment the different interaction between individual BVOC and water molecules may also reduce substantially the total concentration of the volatiles (Kari et al., 2018).BVOC fluctuations observed between consecutive samplings could then be attributed to resulted environmental fluxes in the chamber misleading correct estimation of BVOC levels in the i-RTG. Unfortunately, assumptions verification required additional analysis and eventually the monitoring of physiological parameters (e.g., CO_2 rates, stomatal conductance, etc.) that in our experiment were not conducted. Nevertheless, the reproducibility of the sampling method was assessed. Validation was performed on the blank samples in which no BVOC was detected (levels below the LLOQ) and on the statistical tests on the collected emissions and the environmental records between the two sections of the static enclosure, where indeed no significant differences were highlighted in either case.

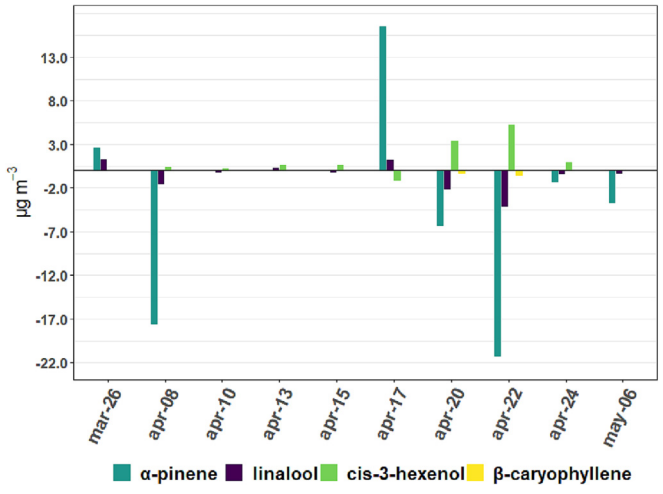
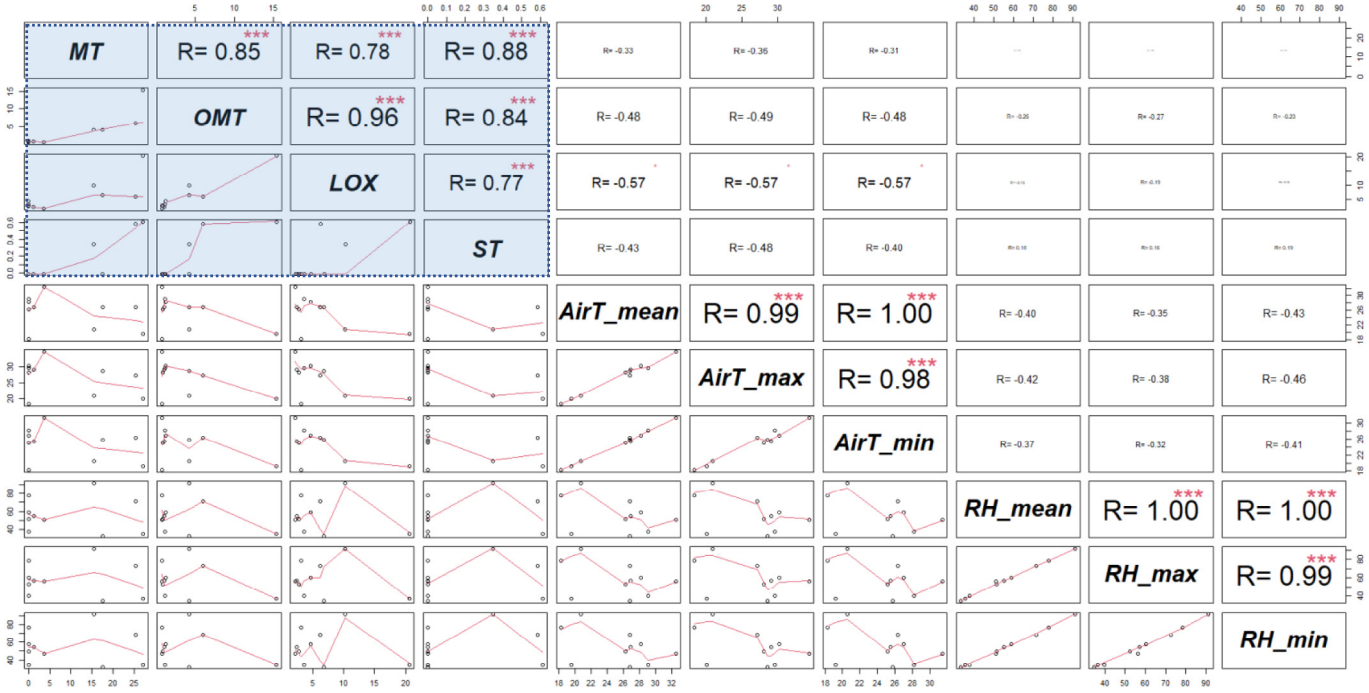
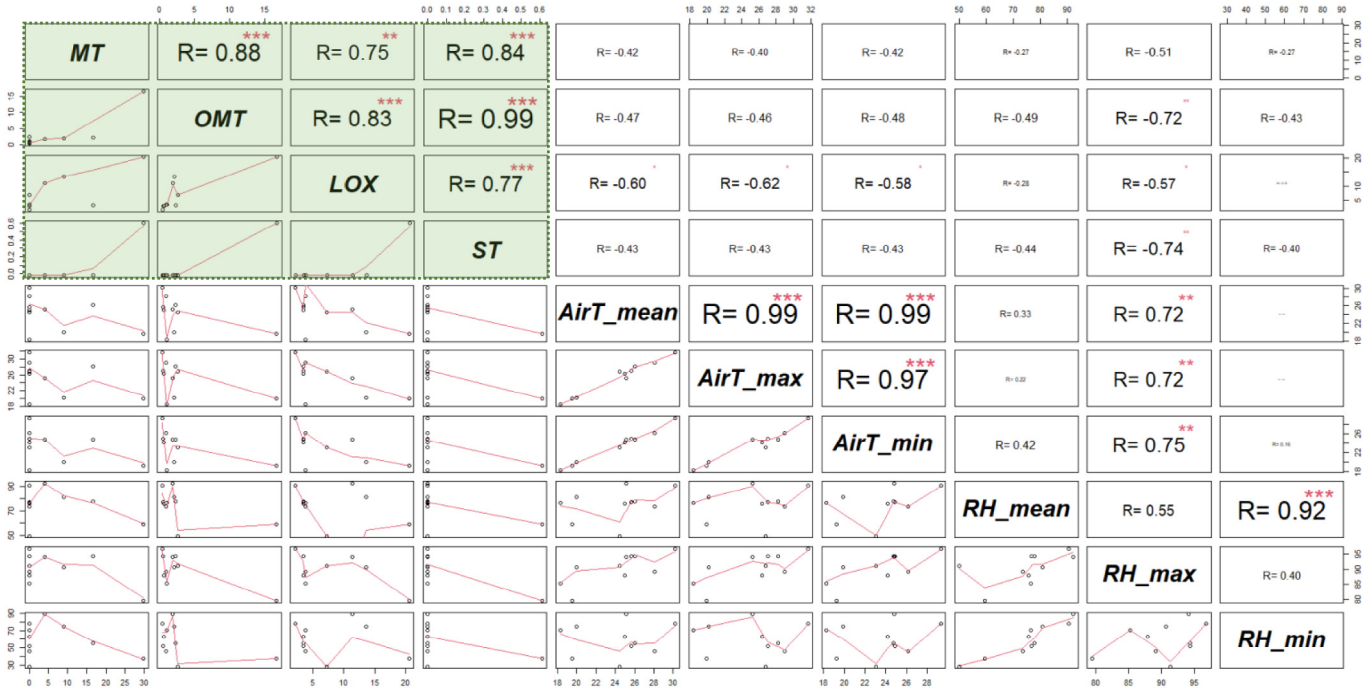


Fig. 6. BVOC emissions (µg m^{-3}) variation after subtraction of the total emissions collected in the empty section from those collected in the plant section.



(A.1)



(A.2)

Fig. 7. Positive and negative Pearson's *r* correlations between environmental variables (mean, maximum and minimum relative humidity, and air temperature: RH_mean, RH_max, RH_min, AirT_mean, AirT_max, AirT_min) and individual BVOC classes (MT monoterpene, OMT oxygenated monoterpene, LOX lipoxygenase derivate and ST sesquiterpene) detected in the chamber during air sampling in the empty (A.1) and plant (A.2) sections; significant correlations $P < 0.001$, $P < 0.01$ and $P < 0.05$ are indicated by the symbols '***', '**' and '*'. Correlations with radiation data are omitted here since they are not significant in any of the cases.

5. Conclusions

According to our findings, expected BVOC emissions released by a leafy crop in an i-RTG can be rationally contained considering low levels found in a small fraction (1 m^3) of it, and more dispersed in relation to traditional indoor systems due to recurrent turnovers of the air enclosed. Regarding the choice of the proper sampling strategy, we suggested dynamic sampling in static enclosure because of an easy-to-implement method for preliminary screening of BVOC emissions, which can provide the indicative levels of BVOC fluxes along a short cultivation cycle (4 months). The occurrence of artifacts during the measurement leading to wrong estimation of current BVOC levels is still higher compared to most recent sampling technologies.

Table 3
Emission factors (average of nearby air measurements) of the reviewed BVOCs over the crop cycle in the i-RTG, and the environmental factors, mean, maximum and minimum temperature ([a], [b], [c]) and relative humidity ([A], [B], [C]) most significantly correlated with them.

BVOC EF (ng g^{-1} h^{-1})				
Plant phenological stage	α-Pinene	Linalool[b, A]	cis-3-Hexenol[a, b, c, A]	β-Caryophyllene[b, A]
Full vegetative	75.852	51.335	78.967	1.558
Reproductive	17.715	4.257	11.286	0.000
Productive	14.294	5.067	20.683	0.000
Senescent	0.000	2.040	11.082	0.000

Bias may be reduced improving the performance of the sampling set-up where the conservation of the environmental conditions is pivotal. This could be achieved for instance by monitoring specific physiological predictors of plant response and adjusting the length of the measurement. We compared levels found with official indoor EU-LCIs values as based on rigorous protocols of exposure degree to a specific volatile. Even at the conditions of the highest emission rates for all detected BVOCs levels were much smaller than given LCIs. However, even in most updated tables (2021) detailed LCIs of BVOCs are limited to one class of compounds, basically monoterpenes, and direct comparison of levels related to other speciates (lipoxygenase derivates and sesquiterpenes) is not possible. Since plants can emit simultaneously a variety of different volatile speciates having each a defined interaction with the environment their potential risks associated to the human health require attention. Their future evaluation may upgrade current knowledge and support the update of reference limits.

CRediT authorship contribution statement

Gaia Stringari: Conceptualization, Methodology, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization. **Joan Villanueva:** Methodology, Validation, Resources, Data curation, Writing – review & editing, Supervision. **Antoni Rosell-Melé:** Conceptualization, Methodology, Resources, Supervision, Funding acquisition. **Nuria Moraleda-Cibrián:** Methodology, Validation, Resources. **Francesco Orsini:** Conceptualization, Methodology, Writing – review & editing, Supervision, Funding acquisition, Project administration. **Gara Villalba:** Conceptualization, Resources, Writing – review & editing, Supervision. **Xavier Gabarrell:** Conceptualization, Methodology, Validation, Writing – review & editing, Supervision, Project administration, Funding acquisition.

Data availability

Data will be made available on request.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2023.162319.

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