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Monoterpenes from tropical forest and oil palm plantation floor in Malaysian Borneo/Sabah – emission and composition

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Abstract (10-15 lines)

Regional estimates of VOC fluxes focus largely on emissions from the canopy and omit potential contributions from the forest floor including soil, litter, and understorey vegetation. Here, we measured monoterpene emissions every two months over two years from logged tropical forest and oil palm plantation floor in Malaysian Borneo using static flux chambers. The main emitted monoterpenes were α -pinene, β -pinene and d-limonene. The amount of litter present was the strongest indicator for higher monoterpene fluxes. Mean α -pinene fluxes were around 2.5-3.5 $\mu\text{g C m}^{-2} \text{ h}^{-1}$ from the forest floor with occasional fluxes exceeding 100 $\mu\text{g C m}^{-2} \text{ h}^{-1}$. Fluxes from the oil palm plantation, where hardly any litter was present, were lower (on average 0.5-2.9 $\mu\text{g C m}^{-2} \text{ h}^{-1}$) and only higher when litter was present. All other measured monoterpenes were emitted at lower rates. No seasonal trends could be identified for all monoterpenes and mean fluxes from both forest and plantation floor were ~100 times smaller than canopy emission rates reported in the literature. Occasional spikes of higher emissions from the forest floor, however, warrant further investigation in terms of underlying processes and their contribution to regional scale atmospheric fluxes.

1. Introduction

Typically, volatile organic compounds (VOCs) are associated with their presence in the atmosphere; more recently they have also been mentioned in connection with soils as a source for biogenic VOCs (Bourtsoukidis et al. 2018; Jardine et al. 2015; Penuelas et al. 2014). Biogenic VOCs are initially degraded in the atmosphere by hydroxyl (OH) radicals which are produced photochemically and responsible for the oxidation of greenhouse gases such as methane (Gray et al. 2010). Biogenic VOCs and some greenhouse gases (e.g. methane) are competitive reactants for available OH radicals and therefore important for predicting the atmospheric lifetime of trace gases. By reducing OH radicals, VOCs can alter atmospheric photochemistry which then results in increasing tropospheric ozone and the production of, for example, organic nitrates (Monson and Holland 2001). Terpenes are highly reactive compounds; in the lower atmosphere their oxidation can lead to the formation of secondary organic aerosols (SOA) which are components of fine particulate matter (PM_{2.5}) and impact on human respiratory and cardiovascular health (Hallquist et al. 2009). Monoterpenes are a class of terpenes that consist of two isoprene units and have the molecular formula C₁₀H₁₆. Terpenoids also include oxygenated monoterpenes (C₁₀H₁₈O) such as eucalyptol and terpenoids often reported in the literature include linalool, geraniol etc. Origins of VOCs include plants, fungi and microbes with abiotic and biotic factors as potential drivers of the fluxes (Penuelas et al. 2014). Important VOC emission sources from the forest floor include leaf, needle and wood litter (Mäki et al. 2019; Šimpraga et al. 2019) as well as root systems of living and dead trees (Lin et al. 2007). Microbial decomposition of soil organic matter has been recognised as the main source of VOC emissions from soil (Leff and Fierer 2008). In forests, plant litter contributes a large proportion to soil organic matter, hence VOC emissions from soils are predominantly associated with the decomposition of plant derived substrates (Penuelas et al. 2014). Furthermore, VOC emissions from soil and litter are thought to be highly variable across litter types and not predictable from measured chemical characteristics of litter

(Gray et al. 2010). In addition, some abiotic processes have been reported to lead to soil VOC emissions, such as evaporation from plant litter (Gray et al. 2010; Greenberg et al. 2012). Soils can also act as sinks for VOCs with microbes using VOCs as carbon source (Albers et al. 2018; Asensio et al. 2012; Greenberg et al. 2012). Soils are complex systems with many processes leading to simultaneous VOC uptake and emissions from the soil and the canopy floor. Hence, there are large uncertainties and knowledge gaps on source and sink strengths of these VOC fluxes (Penuelas et al. 2014). Some studies have reported roots as a strong source for VOCs such as terpenes (Lin et al. 2007), even though it is difficult to separate roots as a source from microbial activity within the soil and above ground. Moreover, it has been reported that soil microorganisms (bacteria and fungi) produce large quantities of diverse volatiles (Schulz and Dickschat 2007). However, the role of monoterpenes and other VOCs in soil ecology is poorly understood (Asensio et al. 2008). A review on soil derived VOC fluxes concludes that emission rates from decomposition processes are much higher than from signalling (communication of plants or microbes to plants or plants and animals) with many functions of VOCs in this context still not understood (Penuelas et al. 2014). Besides, biotic VOC emission rates often exceed those from abiotic controls (Gray et al. 2010). Litter emissions have also been reported to be exponentially dependent on temperature with moisture playing a minor role (Greenberg et al. 2012; Hayward et al. 2001). Litter age appears to be important in determining the magnitude of VOC fluxes (Aaltonen et al. 2011). Previously, impacts of litter VOCs on soil nutrient levels and bacterial community structure have been found to be negligible (Ramirez et al. 2010). At present, the source and sink capabilities of soils are not specifically considered in global VOC estimates from the terrestrial biosphere (Tang et al. 2019).

Most of the published studies on soil VOC fluxes have been carried out in Temperate, Boreal or Mediterranean climates. Data from the Tropics are very limited in the literature, apart from

a recent study on sesquiterpene fluxes from the Amazon rainforest (Bourtsoukidis et al. 2018). Model estimates of the emissions of VOCs from these non-tropical regions predict strong responses to the strong annual cycles of foliar biomass, light intensity and temperature. In contrast, tropical regions stand out as a dominant source year round due to constant temperature and light levels, and little variability in foliage biomass of deciduous trees (Kuhn et al. 2004). Due to the high productivity of tropical ecosystems, the activity of soils could potentially provide a greater contribution to atmospheric VOCs than in colder climates and this could also be seen within canopy level fluxes (Penuelas et al. 2014). Tropical studies available in the literature are predominantly reporting canopy fluxes from the Amazonian region (Alves et al. 2016; Kesselmeier et al. 2000; Kuhn et al. 2004; Yáñez-Serrano et al. 2018). Biogenic VOC emissions from vegetation represent a substantial carbon loss for plants and significantly contribute to the carbon balance of terrestrial ecosystems. This is especially true for the Tropics (Guenther 2002; Kesselmeier et al. 2002) where the magnitude of VOC losses from soil and litter, in relation to the carbon budget, is less clear.

Canopy VOC emissions from oil palm (OP) plantations are poorly understood but have been reported to be higher than from primary forests (Fowler et al. 2011; Hewitt et al. 2009). In their study, emissions from oil palm consisted mainly of isoprene whilst canopy emissions from the tropical forest in South East Asia (Borneo) were dominated by monoterpenes (Fowler et al. 2011). Canopy scale emissions from OP plantations, especially isoprene (Misztal et al. 2011; Wilkinson et al. 2006), have received attention in the past due to their impact on air quality; however, soil emissions of monoterpenes, or the contribution of soil emissions to total fluxes, are generally not considered.

Deforestation and forest degradation in Southeast Asia, to a large degree, has happened for establishing OP plantations (Gaveau et al. 2016; Lee-Cruz et al. 2013; Wilcove et al. 2013). In Malaysia, Indonesia and Papua New Guinea the area covered with industrial OP plantations has increased rapidly in recent decades, from 3.5 Mha in 1990 to 13.1 Mha in 2010, of which 4.1% of the land was undisturbed forest and 32.4% was disturbed forest before conversion (RSPO 2013). In 2000, 88% (20.8 Mha) of the land was covered by natural forest in Malaysia, by 2010 this had decreased to 69% (16.6 Mha) and 91% of the deforestation resulted in complete tree cover loss (Global Forest Watch 2018). Land-use change does not only change canopy VOC emission rates, but can potentially have a large impact on microbially derived VOC emissions from litter decomposition (Gray et al. 2010). It has been recognised that more long-term measurements are needed to better characterise seasonal and interannual variability to estimate present and future impact of biogenic VOC fluxes (Alves et al. 2016; Kuhn et al. 2004) and this should be the case not only for studying fluxes from the canopy, but also from the soil and plant litter.

The objective of this scoping study is to broadly characterise the magnitude and composition of VOC emissions from logged forest and oil palm plantation floor as well as from a small riparian area adjacent to one OP plantation in Malaysian Borneo, Sabah. The focus of this study was on potential VOC sources, hence only monoterpenes were considered as soils are more likely a sink for isoprene (Carrión et al. 2020).

2. Methods

2.1 Site description

Measurements took place during 2015 and 2016 within the Stability of Altered Forest Ecosystems (SAFE) project in Malaysian Borneo (4°49'N, 116°54'E). The SAFE project was

set up in Sabah in 2011 in a secondary forest area designated by the Sabah government for conversion to oil palm (OP) plantations with the primary aim to study how habitat fragmentation affects the forest ecosystem (Ewers et al. 2011). The forest was selectively logged for dipterocarps first in the 1970s then for a second time between 2000 and 2008. In this study, we have chosen 3 forest locations and 3 OP locations of different ages (2, 7 and 12 years) as well as a small riparian area. All OP plantations were on terraced soil. The soils at SAFE are classed as orthic Acrisols or Ultisols (Riutta et al. 2018). The climate is wet tropical with a wet season typically from October to February and a dry season typically from March to September, although seasons are not as pronounced as in other tropical regions. Regional average monthly temperatures are 32.5°C and regional mean monthly rainfall is 164.1 mm (climate-data.org, 2019).

2.2 Monoterpene flux measurements

For soils, enclosure chambers are the most widely used sampling technique due to its suitability of all types of terrain (Penuelas et al. 2014). We measured monoterpene fluxes from 4 chambers in the 3 logged forests (LF, B, and E). In the OP plantations, monoterpene fluxes were measured from 6 chambers in a 7-year old oil palm plantation (OP7), 4 in a ~2-year old plantation (OP2) and 4 in a 12-year old oil plantation (OP12). In addition, we sampled 2 chambers in a riparian area adjacent to OP7. For exact GPS locations see published dataset (Drewer et al. 2020a). At each site chambers were within a few tens metre squared. Flux measurements were made from all 28 chambers every two months over a two-year period from January 2015 to November 2016, resulting in 12 measurement occasions for each of the chambers and a total of 336 individual flux measurements.

162 Opaque PVC soil chambers, as previously used in studies measuring monoterpenes from soil
163 and litter (Asensio et al. 2007; Greenberg et al. 2012), consisted of a collar that stayed in the
164 ground for the duration of the 2-year measurement period and a lid that was tightly fastened
165 during sampling only (Drewer et al. 2020b; Drewer et al. 2017). The 40 cm diameter collars
166 were inserted into the ground without disturbing litter or removing ground vegetation to capture
167 natural conditions within the 0.1257 m² area. The chamber volume including lids was
168 approximately 30 L. Sample lines (6 mm PTFE tubing) were inserted through the chamber lids
169 and attached to a hand pump (210-1003MTX, SKC Ltd, Blandford Forum, UK) drawing air
170 from inside the chamber at a flow rate of 200 mL min⁻¹ through a 6 mm OD stainless steel
171 absorbent cartridge. ‘Clean’ air (stripped of sampled VOCs) was cycled back into the chamber,
172 which also ensured air movement in the chambers, and hence no fan was required. No ozone
173 filter was used during our study although measurements from a previous study conducted using
174 static forest floor chambers with and without an ozone filter resulted in no differences in VOC
175 emissions (Hellén et al. 2006). The cartridges used in this study were packed with 200 mg
176 Tenax® TA 60/80 and 100 mg Carbotrap® 20/40 (20273 SUPELCO, Sigma-Aldrich). At the
177 same time, ambient air was sampled outside the chamber via a PTFE sample line positioned
178 directly above the chamber and connected to a hand pump. Ambient air and chamber air were
179 pumped concurrently for about 25 min resulting in a 5 L sample. Cartridges were kept
180 refrigerated and sent to UK CEH for analysis typically one to two months after sampling. This
181 length of time of storage has been deemed acceptable regarding the stability of the compounds
182 of interest (Helin et al. 2020). The samples were analysed using gas chromatography-mass
183 spectrometry (Clarus 500, Perkin Elmer, Wellesley, MA, USA) with a two-stage automatic
184 thermal desorption unit (ATD 400, Perkin-Elmer, Wellesley, MA, USA). The cartridges were
185 desorbed at 280 °C for 6 min under a flow of helium with subsequent trapping onto a Tenax®
186 TA cold trap at -30 °C. The second stage of desorption was achieved by flash heating the cold

trap to 300 °C for 6 min to flush the sample through a heated transfer line (200 °C) onto the GC column (Ultra-2 column, 100 m length, 0.2 mm I.D., 5% phenylmethyl silica, Agilent, Palo Alto, CA, USA). The GC oven was held at 35 °C for 2 min, ramped to 160 °C at 3 °C min⁻¹ then ramped to 280 °C at 45 °C min⁻¹ before being held at 280 °C for 10 min (Morrison et al. 2016; Purser et al. 2020b). The compounds were then detected using a tuned mass spectrometer (Perkin Elmer, Wellesley, MA, USA) operating in total ion count mode (Morrison et al. 2016; Purser et al. 2020b).

Ion m/z 93 was selected for quantification of monoterpenes. Quantification was performed by comparison with calibrations using standards of monoterpenes measured at the start and end of each sample run as well as after every 6 samples. Monoterpene standards were prepared from a mixed stock solution of the following monoterpenes at a concentration of 3 ng µL⁻¹ diluted in methanol and contained α-pinene, β-pinene, d-limonene, eucalyptol, 3-carene, and camphene. Aliquots of the mixed monoterpene stock solution were pipetted directly onto cartridges (the same as used for field sampling) under a flow of helium. Peaks in sample chromatograms were identified by comparison to the internal library of the GC-MS (National Institute of Standards and Technology (NIST)) and by comparison with the retention time of the standard. Peak areas were used to quantify monoterpene concentrations in the samples.

Limit of detection (LoD) for each analyte was calculated using repeated blank measurements and were as follows: α-pinene 0.78 ng, β-pinene 0.90 ng, d-limonene 0.60 ng, 3-carene 0.94 ng, eucalyptol 1.76 ng, camphene 0.92 ng (Purser et al. 2020b).

Monoterpene fluxes from the forest floor (F_{floor}) (µg C m⁻² h⁻¹) were calculated using Equation (1), where C_{sample} is the concentration of a monoterpene inside the chamber (µg C L⁻¹), C_{ambient}

is the concentration of a monoterpene in the ambient air outside the chamber ($\mu\text{g C L}^{-1}$), A is the area of forest floor inside the chamber (m^2), V is the volume inside the chamber (L), and t is the sampling duration (min) (Purser et al. 2020a).

$$F_{\text{floor}} = \frac{[C_{\text{sample}} - C_{\text{ambient}}] \times V \times 60}{A \times t}$$

Equation 1

Uncertainties of the individual calculated fluxes were 17% for monoterpenes which was derived by an error propagation published in Purser et al. (2020b).

Monoterpene emission rates from foliage are commonly normalised to a temperature of 30 °C based on empirically derived coefficients (Guenther et al. 1993). These formulas have also been applied to normalise emissions from the forest floor (Hayward et al. 2001). It is likely that both abiotic and biotic factors act as drivers for monoterpene emissions from the forest floor (Penuelas et al. 2014) and would warrant further investigation as to whether the algorithms are applicable or not. This, however, was not possible in our study due to the limited range of environmental conditions over which the data was collected. In addition, we occasionally also measured negative fluxes (i.e. uptake) for which the algorithms would not be appropriate and we consequently decided not to attempt to normalise fluxes in this case.

As measurements were carried out *in situ*, it was not possible to differentiate between monoterpene emissions from roots, soil or decomposing litter as well as microbial sources. Therefore, reported fluxes are net fluxes comprising all sources from forest and oil palm plantation floor.

2.3 Soil and litter measurements

A handheld Omega HH370 temperature probe (Omega Engineering UK Ltd., Manchester, UK) was used to measure soil and air temperatures at each chamber location at a soil depth of 10 cm and by positioning the temperature sensor 30 cm above the soil surface at chamber height. A portable probe (Hydrosense 2; Campbell Scientific, Loughborough, UK) was used to measure volumetric soil moisture content (VMC) at a depth of 7 cm.

To measure soil physicochemical parameters, soil cores were taken from the top 10 cm next to the chambers and on the last sampling occasion from within the chambers. For each chamber, soil pH was measured from the top 0-10 cm on three occasions: one close to the chamber at the start of the measurement period, a second close to the chamber after two months, and the third was taken inside the chamber after the last flux measurement. For pH measurements, 10 g of fresh soil was mixed with deionised H₂O (ratio 1:2), and after 1 hour were analysed on a MP 220 pH meter (Mettler Toledo GmbH, Schwerzenbach, Switzerland). Soil samples for bulk density were collected from inside each chamber after the final flux measurement. Galvanised iron rings (98.17 cm³) with a sharp edge were inserted in the upper soil layer with a hammer to 5 cm depth without compaction. Samples were oven-dried at 105°C until constant weight (usually 48 hours) and bulk density (g cm⁻³) was calculated based on the dry weight occupying the volume of the ring.

Total C and N in soil and litter was measured once on the last sampling occasion. Soil samples were taken from the top 0-10 cm inside the chambers. The samples were air dried in the field laboratory and a subsample of each was dried at 105°C to constant weight to convert the results to oven-dry weight. They were then ground and analysed at the Forest Research Centre in Sandakan on an elemental analyser (Vario Max CN Elemental Analyzer (Elementar Analysensysteme, Germany)). Litter was collected from the surface area of each chamber

(0.1257 m²) on the last sampling occasion and air dried at 30 °C and analysed for total C and N as described above.

2.4 Data analysis

Minitab® 17.3.1 software was used for data analysis and descriptive statistics. The dataset of measured monoterpenes and associated soil physicochemical parameters is published in the SAFE zenodo database (Drewer et al. 2020a).

3. Results and Discussion

At SAFE, the mean monthly rainfall during the two years of study period (2015 and 2016) was 190 mm, ranging from 45 mm during the driest month (Mar 2015) and 470 mm during the wettest month (Sep 2016). Annual rainfall was 1927 mm in 2015 and 2644 mm in 2016 (Drewer et al. 2020b) with 2015 being an unusually dry year. Mean air temperature over the two years of field measurements was 25.8 °C (standard error ± 0.1 °C) in the logged forest, 29.0 °C (± 0.2 °C) in the oil palm plantations and 29.6 °C (± 0.5 °C) in the riparian area. Soil temperature was constant throughout the year and averaged 24.5 °C (± 0.1 °C) for logged forest, 26.6 °C (± 0.1 °C) for OP and 26.8 °C (± 0.2 °C) in the riparian area. Mean volumetric soil moisture content was 25.7% ($\pm 0.9\%$) in logged forest, 25.3% ($\pm 0.6\%$) in oil palm plantations and 30.3% ($\pm 1.0\%$) in the riparian area. No direct correlations with temperature or moisture and emitted monoterpenes could be established. This may be because temperature is almost constant throughout the year and wet and dry seasons are not very pronounced in Sabah.

Soil physicochemical parameters are shown in Table 1. These are a subset of data published in Drewer et al. (2020b), as only half of the locations were sampled for monoterpenes; hence, the

data in Table 1 are slightly different to the data in Drewer et al. (2020b). However, the overall differences between sites were broadly the same. Soil pH was lower from forest site B (pH 3.9) than from any of the other forest sites E and LF (pH 6.4 and 6.1) with OP plantations between pH 4.6-4.7 and pH 5.6 in the riparian area (Table 1). Bulk density was higher in OP and the riparian area ($\sim 1.3 \text{ g cm}^{-3}$) compared to the forests ($\sim 0.8 \text{ cm}^{-3}$), possibly due to compaction caused by using heavy machinery for clearing, terracing and planting oil palms. Total soil N and C were higher in the forest soils than the plantation and riparian soils ranging from 0.3-0.5% N in forests versus 0.04 to 0.1% N in plantation and riparian areas; and 4-9% C in forests versus 0.5-1% C in plantation and riparian areas. Contrary, total C and total N content of the litter was similar at all sites and for all land-uses (~ 1.6 - 1.9% N and 32-42% C). The amount of litter present was very variable. The main difference between the logged forest and the oil palm plantations was the total amount of litter present inside the chambers. All chambers installed in the forests contained litter. In contrast, chambers in the oil palm plantations had no or very little litter. None of the OP12 chambers contained litter at all, in OP7 only one chamber had litter present and in OP2 two of the four chambers had litter (on average 72 g dry weight). The riparian area was to a large extent covered by ground vegetation and therefore did not have a large amount of litter present either (on average 16 g dry weight). Forest chambers had litter between 50 and 130 g dry weight with a high variability even within sites. Litter samples were only taken after the last measurement occasion as to not disturb ongoing flux measurements. However, according to our own visual inspection at every sampling occasion, we do believe that the samples taken were representative of the location throughout the 2-year sampling period. Scaled to 1 m^2 , mean litter weights (with the range in parentheses) in the different land-uses were 747 (187 - 1615) g m^{-2} in the logged forests, 430 (0 - 1070) g m^{-2} in the oil palm plantations and 125 (103 – 147) g m^{-2} in the riparian area.

Generally, mean monoterpene fluxes were low at all sites and showed high variability (expressed as the minimum and maximum) (Table 2). Averaging over all measurement occasions, proportionally α -pinene, β -pinene and d-limonene were emitted as the highest fluxes of all measured monoterpenes at all sites, with exception of OP7 and OP12, where eucalyptol had a higher proportion than β -pinene (Figure 1). The 3 measured monoterpenes that were present at every site and every measurement occasion were α -pinene, β -pinene and d-limonene (Table 2, Figure 2). In contrast, 3-carene was only present in some months and camphene and eucalyptol were not present in 2015 at all, only in 2016 when the soils were slightly wetter (Table 2, Figure 2).

Mean emissions for α -pinene from the logged forest floor were $2.25 \mu\text{g C m}^{-2} \text{ h}^{-1}$ (min and max: -0.16 and $47.39 \mu\text{g C m}^{-2} \text{ h}^{-1}$) for site B, 2.76 (-0.42 to 85.35) $\mu\text{g C m}^{-2} \text{ h}^{-1}$ for site E and 3.48 (-0.05 to 124.42) $\mu\text{g C m}^{-2} \text{ h}^{-1}$ for site LF. Minimum and maximum fluxes highlight the large variability even within one site (Table 2). Mean α -pinene fluxes from the oil palm plantation floor were overall lower, giving values of: 2.87 (-0.43 to 56.31) $\mu\text{g C m}^{-2} \text{ h}^{-1}$ at OP2, 0.45 (-0.11 to 3.65) $\mu\text{g C m}^{-2} \text{ h}^{-1}$ at OP7, 1.15 (-0.17 to 10.66) $\mu\text{g C m}^{-2} \text{ h}^{-1}$ at OP12, and 2.78 ($-0.$ to 29.6) $\mu\text{g C m}^{-2} \text{ h}^{-1}$ in the riparian area. Mean fluxes for β -pinene were $0.22 - 0.5 \mu\text{g C m}^{-2} \text{ h}^{-1}$ from the three forest sites, 0.25 to $0.3 \mu\text{g C m}^{-2} \text{ h}^{-1}$ in OP12 and OP7, $2.78 \mu\text{g C m}^{-2} \text{ h}^{-1}$ in OP2 (largely driven by 2 exceptionally high points in one day), and $1.3 \mu\text{g C m}^{-2} \text{ h}^{-1}$ in the riparian area; details can be found in Table 2. Mean emissions for the third most important monoterpene, d-limonene, were 0.54 to $1.27 \mu\text{g C m}^{-2} \text{ h}^{-1}$ from the forest sites, 0.6 to $1.95 \mu\text{g C m}^{-2} \text{ h}^{-1}$ in the plantations and $1.77 \mu\text{g C m}^{-2} \text{ h}^{-1}$ in the riparian area. Fluxes for the other measured compounds (3-carene, camphene and eucalyptol) were lower and are listed in Table 2. We calculated total monoterpene emissions from these six main monoterpenes, as any other monoterpenes present in the samples were of non-significant quantities, in order to put our

results into context with the literature where often only total monoterpene emissions reported. Generally, mean total monoterpene fluxes were low at all sites with the occasional ‘spike’, especially for the forest sites and OP2. Total monoterpene emissions from forest site B were slightly lower (mean as well as maximum fluxes) than the other two forest sites. Forest site B had been identified as the site with the lowest soil pH and a different bacterial community than the other forest sites and all oil palm plantations sites (Drewer et al. 2020b). As microbial diversity was only measured a couple of times during the 2-year measurement period, no direct correlation with monoterpene fluxes could be determined. The variability of a given monoterpene for a given site was very high and there was no discernible temporal trend (Figure 2).

OP2, which showed higher monoterpene fluxes than OP7 and OP12, had more litter present than the other two plantations. A study also carried out in the SAFE area, similarly concluded that the presence of litter *per se* was more important for maintaining soil microbial processes than litter quality or diversity (Kerdraon et al. 2020). This links in with our findings that the presence of litter was the main indicator for higher monoterpene fluxes. OP2 was the youngest plantation sampled here, had no closed canopy cover yet and more weeds as understory vegetation which might have contributed to the higher litter amount compared to the older plantations. A few laboratory studies have found that biotic processes and sources of monoterpenes are more important than abiotic ones (Gray et al. 2010; Leff and Fierer 2008). Tropical regions generally show less seasonality due to low oscillations in temperature and light intensity compared to temperate regions. This may explain the lack of direct correlations and lack of distinct temporal variability in our study.

Very few (field) studies have reported forest floor VOC fluxes; instead, they quote concentrations/mixing ratios or emissions expressed per dry weight of foliage, but not the surface area. Therefore, it is difficult to put our measurements into context. Comparing soil and foliar monoterpene emissions from Sitka Spruce in the UK established that on a land area basis, soil emissions were relatively insignificant when compared with tree monoterpene emissions (Hayward et al. 2001). The magnitude of their published emission rate from soil including litter (converted to carbon equivalent) was a mean of $\sim 30 \mu\text{g C m}^{-2} \text{ h}^{-1}$. The magnitude of our measured forest and oil palm plantation floor fluxes were about ten times lower. In Boreal forest, total monoterpene emissions ranged from ~ 17 to $50 \mu\text{g C m}^{-2} \text{ h}^{-1}$ (Mäki et al. 2019), which again is overall higher than what we measured. This might be due to the higher proportion of coniferous trees or higher amounts of litter present in more organic rich soil. Fluxes of individual monoterpenes in a Boreal forest were on average $2.6 \mu\text{g C m}^{-2} \text{ h}^{-1}$ for α -pinene, $0.17 \mu\text{g C m}^{-2} \text{ h}^{-1}$ for β -pinene and $0.01 \mu\text{g C m}^{-2} \text{ h}^{-1}$ for d-limonene (Aaltonen et al. 2011) which is slightly lower than our measured fluxes. In a Mediterranean shrubland, the dominant measured monoterpenes from *Pinus* were α -pinene, β -pinene, d-limonene and camphene (Asensio et al. 2008), which are the same dominant compounds as in our study and the authors suggest roots were the likely source of these (Lin et al. 2007).

In an Amazonian tropical forest, canopy VOC fluxes were measured as $0.20 \text{ mg C m}^{-2} \text{ h}^{-1}$ for α -pinene, and $0.39 \text{ mg C m}^{-2} \text{ h}^{-1}$ for the sum of monoterpenes (Kuhn et al. 2007) which is on average ~ 100 times higher than from our forest floor measurements. Average midday monoterpene fluxes from Amazonia were also reported as $\sim 1 \pm 0.5 \text{ mg C m}^{-2} \text{ h}^{-1}$ (Karl et al. 2007) derived from airborne fluxes. Alves et al. (2016) summarised published canopy fluxes spanning a range of different measurement and modelling techniques from the Amazon and the sum of total monoterpenes ranged roughly from ~ 0.2 to $\sim 2 \text{ mg C m}^{-2} \text{ h}^{-1}$ with the high end

often representing midday fluxes rather than daily averages. Similar magnitudes from canopy fluxes were reported from Southeast Asia. The field campaign by Fowler et al. (2011) conducted in Malaysia Borneo, reports total monoterpene canopy fluxes from tropical forest to be higher than from OP, with forest canopy fluxes peaking at midday around $0.4 \text{ mg m}^{-2} \text{ h}^{-1}$ and OP fluxes around zero, ranging from $(-0.2 \text{ to } 0.2 \text{ mg m}^{-2} \text{ h}^{-1})$. The authors report the dominant VOC emitted being isoprene, with emissions from OP five times higher than from the forest. Monoterpenes comprised 18% of the rainforest canopy fluxes and less than 1% in the OP plantation. Our mean measured total monoterpene fluxes from forest floor are ~100 times smaller for both oil palm plantations and forest sites. However, maximum fluxes measured as occasional spikes are only ~5-10 times lower.

Most regional and global estimates of VOC budgets so far only include emissions from vegetation, although some laboratory and field studies have indicated the importance of VOC fluxes from soils (Tang et al. 2019). In this study, we have provided a large dataset of measured monoterpene fluxes over a 2-year period, which can be used as quantitative information for tropical forest and oil palm plantation floor emissions. We did not find differences in monoterpene emissions related to dry and wet seasons suggesting monoterpene emissions from the forest floor in this region are more consistent unlike other rainforest regions. The composition of measured monoterpenes in our study is also comparable to previously published studies from Temperate or Boreal regions. We conclude that although monoterpene emissions from the forest and plantation floor are on average 100 times smaller than from the canopy, they warrant further investigation as maximum measured fluxes were only ~5-10 times lower than reported canopy fluxes. Therefore, drivers of these emission spikes, for example, microbial activity and diversity, warrants more detailed investigation.

Conclusions

Fluxes of monoterpenes from forests and oil palm plantation floor and a small riparian area in Sabah, Malaysia were ~100 times smaller compared to published canopy fluxes within the same region, with maximum 'spikes' only ~5-10 times smaller than the published canopy fluxes. The amount of litter present was the strongest contributing factor towards monoterpene fluxes rather than land-use *per se*. The dominant measured monoterpenes were α - and β -pinene and to a lesser extend d-limonene. In light of potential land-use change, it is important to establish emissions rates from existing land-uses to be able to make predictions on future monoterpene fluxes and their potential impact. Process-orientated measurements are needed for model parameterisation to enable models to assess the contribution of ground VOC fluxes towards climate change and air quality.

Table 1. Soil and litter physicochemical parameters from soil chambers at 7 sites. B, E, LF = logged forest, OP2, OP7, OP12 = oil palm plantations of different stand ages, RR = riparian reserve over the two-year measurement period (Jan 2015 to Nov 2016). N = number of individual chambers/measurements.

Variable	site	N	Mean	SE	StDev	Median
<i>pH</i>	B	4	3.87	0.07	0.14	3.89
	E	4	6.38	0.39	0.79	6.42
	LF	4	6.10	0.33	0.66	6.37
	OP2	4	4.67	0.07	0.13	4.69
	OP7	7	4.71	0.08	0.21	4.77
	OP12	4	4.59	0.06	0.12	4.57
	RR	2	5.57	0.62	0.87	5.57
<i>Bulk density [$g\ cm^{-3}$]</i>	B	4	0.80	0.06	0.11	0.79
	E	4	0.79	0.14	0.27	0.81
	LF	4	0.73	0.08	0.17	0.67
	OP2	4	1.27	0.06	0.13	1.26
	OP7	7	1.26	0.08	0.22	1.38
	OP12	4	1.27	0.05	0.09	1.29
	RR	2	1.28	0.12	0.16	1.28
<i>Soil N [%]</i>	B	4	0.33	0.03	0.07	0.34
	E	4	0.48	0.18	0.35	0.48
	LF	4	0.29	0.08	0.15	0.26
	OP2	4	0.04	0.00	0.01	0.04
	OP7	7	0.09	0.02	0.06	0.06
	OP12	4	0.08	0.02	0.03	0.07
	RR	2	0.10	0.05	0.07	0.10
<i>Soil C [%]</i>	B	4	5.00	0.56	1.11	5.00
	E	4	9.24	4.50	9.00	9.08
	LF	4	4.04	1.22	2.43	3.39
	OP2	4	0.55	0.03	0.06	0.56
	OP7	7	1.04	0.20	0.53	0.93
	OP12	4	0.78	0.08	0.16	0.78
	RR	2	0.95	0.20	0.28	0.95
<i>C/N soil</i>	B	4	15.36	0.44	0.88	15.68
	E	4	14.98	3.88	7.76	15.00
	LF	4	13.91	1.23	2.47	14.50
	OP2	4	14.58	0.34	0.69	14.50
	OP7	7	12.70	1.23	3.24	13.00
	OP12	4	10.27	1.12	2.24	10.69
	RR	2	11.33	3.67	5.19	11.33

<i>litter dry weight [g]</i>	B	4	132.40	27.80	55.50	123.10
	E	4	100.50	13.30	26.70	94.30
	LF	4	48.80	13.30	26.60	43.00
	OP2	2	71.90	62.60	88.50	71.90
	OP7	1	18.50	*	*	18.50
	OP12	0	*	*	*	*
	RR	2	15.70	2.80	3.96	15.70
<i>Litter N [%]</i>	B	4	1.54	0.14	0.27	1.46
	E	4	1.88	0.02	0.05	1.88
	LF	4	1.60	0.20	0.40	1.63
	OP2	2	1.82	0.07	0.10	1.82
	OP7	1	1.54	*	*	1.54
	OP12	0	*	*	*	*
	RR	2	1.99	0.00	0.00	1.99
<i>Litter C [%]</i>	B	4	34.84	2.84	5.69	34.03
	E	4	41.83	1.59	3.18	41.77
	LF	4	34.29	3.84	7.68	34.49
	OP2	2	41.34	7.69	10.87	41.34
	OP7	1	31.99	*	*	31.99
	OP12	0	*	*	*	*
	RR	2	42.96	2.08	2.95	42.96

* No litter present at OP12 and only in one chamber at OP7

Table 2. Monoterpene (MT) fluxes [$\mu\text{g C m}^{-2} \text{ h}^{-1}$] from soil chambers at 7 sites. B, E, LF = logged forest, OP2, OP7, OP12 = oil palm plantations of different stand ages, RR = riparian reserve over the two-year measurement period (Jan 2015 to Nov 2016). N = number of individual measurements. Total MT (total monoterpenes) = sum of all measured monoterpenes.

MT flux [$\mu\text{g C m}^{-2} \text{ h}^{-1}$]	site	N	Mean	SE	StDev	Min	Median	Max
<i>α-pinene</i>	B	48	2.25	1.07	7.40	-0.16	0.38	47.39
	E	48	2.76	1.77	12.26	-0.42	0.36	85.35
	LF	48	3.48	2.58	17.91	-0.05	0.21	124.42
	OP2	47	2.87	1.22	8.36	-0.43	0.54	56.31
	OP7	72	0.45	0.08	0.68	-0.11	0.15	3.65
	OP12	48	1.15	0.32	2.22	-0.17	0.32	10.66
	RR	24	2.78	1.40	6.85	-0.11	0.51	29.62
<i>β-pinene</i>	B	48	0.45	0.24	1.68	-0.14	0.05	11.56
	E	48	0.22	0.08	0.57	-0.05	0.05	2.72
	LF	48	0.50	0.22	1.51	-0.02	0.05	9.90
	OP2	47	2.78	2.03	13.89	-0.33	0.15	95.46
	OP7	72	0.30	0.11	0.92	-0.26	0.08	6.65
	OP12	48	0.25	0.06	0.44	-0.05	0.11	2.55
	RR	24	1.30	0.81	3.96	-0.04	0.26	19.64
<i>d-limonene</i>	B	48	0.54	0.15	1.05	-3.26	0.22	4.60
	E	48	1.19	0.23	1.61	-0.11	0.53	6.07
	LF	48	1.27	0.35	2.41	0.00	0.42	12.48
	OP2	48	1.95	0.54	3.76	-0.01	1.15	23.60
	OP7	72	0.60	0.10	0.88	-0.26	0.29	5.15
	OP12	48	1.09	0.25	1.74	-0.16	0.28	8.13
	RR	24	1.77	0.83	4.07	0.00	0.82	20.31
<i>3-carene</i>	B	48	0.09	0.04	0.29	0.00	0.00	1.42
	E	48	0.03	0.01	0.10	-0.15	0.00	0.42
	LF	48	0.18	0.07	0.47	0.00	0.00	2.31
	OP2	48	0.29	0.11	0.79	0.00	0.00	3.47
	OP7	72	0.05	0.01	0.11	-0.07	0.00	0.49
	OP12	48	0.13	0.07	0.51	-0.01	0.00	3.47
	RR	24	0.17	0.11	0.53	-0.04	0.00	2.61
<i>camphene</i>	B	48	0.01	0.02	0.13	-0.42	0.00	0.73
	E	48	0.00	0.00	0.02	-0.10	0.00	0.09
	LF	48	0.03	0.01	0.10	-0.04	0.00	0.53
	OP2	48	0.07	0.05	0.32	-0.30	0.00	1.96
	OP7	72	0.00	0.00	0.03	-0.09	0.00	0.17
	OP12	48	0.09	0.08	0.54	-0.03	0.00	3.74

	RR	24	0.27	0.15	0.75	-0.03	0.00	3.28
<i>eucalyptol</i>	B	48	0.06	0.03	0.19	-0.39	0.00	0.96
	E	48	1.70	1.37	9.49	0.00	0.00	65.60
	LF	48	0.48	0.26	1.83	0.00	0.00	11.86
	OP2	48	0.98	0.36	2.49	0.00	0.00	14.28
	OP7	72	0.69	0.22	1.89	0.00	0.00	12.16
	OP12	48	0.78	0.29	2.01	0.00	0.00	11.34
	RR	24	0.77	0.35	1.73	0.00	0.00	6.13
Total MT	B	48	3.39	1.36	9.41	-3.41	1.04	60.96
	E	48	5.90	2.25	15.55	0.16	1.67	87.10
	LF	48	5.93	2.96	20.52	0.03	1.53	141.92
	OP2	48	8.82	3.64	25.23	0.18	2.77	175.37
	OP7	72	2.09	0.37	3.14	-0.05	1.20	20.94
	OP12	48	3.50	0.68	4.68	-0.16	1.58	23.18
	RR	24	7.06	3.17	15.54	0.05	1.90	73.78

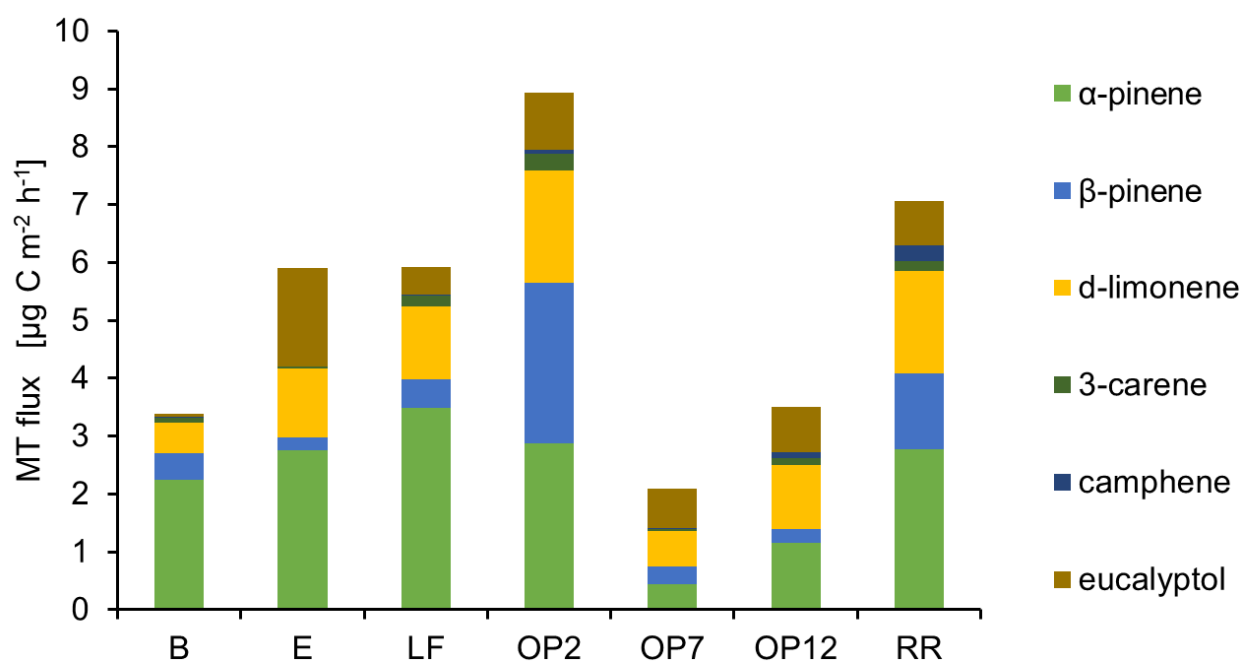


Figure 1. Composition of mean monoterpene (MT) fluxes [$\mu\text{g C m}^{-2} \text{ h}^{-1}$] over the 2-year measurement period (Jan 2015 to Nov 2017) from the different sites (B, E, LF = logged forest, OP12, OP2, OP7 = oil palm plantations of different stand ages, RR = riparian reserve)

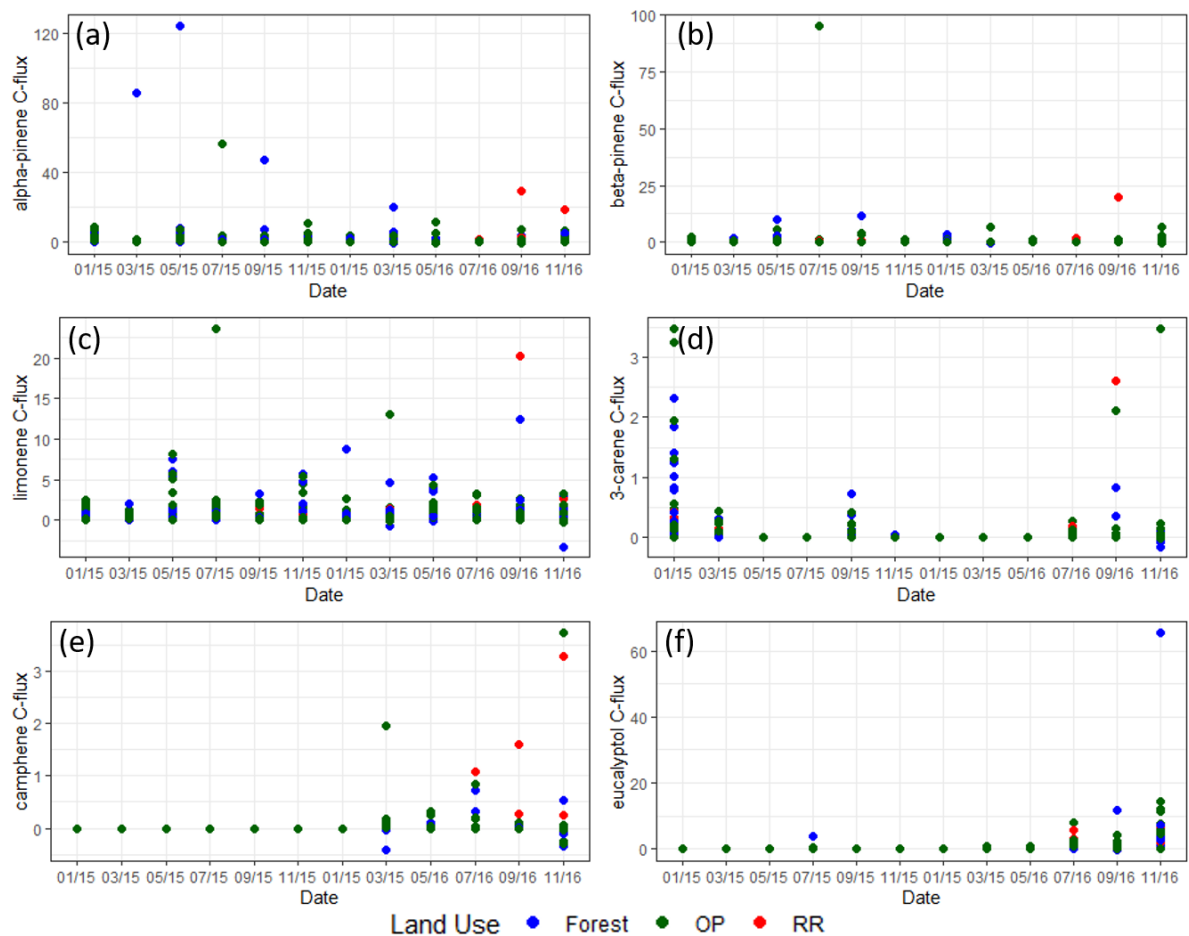


Figure 2. Monoterpene fluxes [$\mu\text{g C m}^{-2} \text{ h}^{-1}$] from soil chambers in logged forest (blue), oil palm plantations (green) and riparian reserve (red) measured every two months from Jan 2015 to Nov 2016. Please note different y-axes scales.

Declarations

Ethics approval and consent to participate

Not applicable

Consent for publication

Not applicable

Availability of data and materials

The dataset used in the present study has been published as:

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Competing interests

The authors declare that they have no competing interests.

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Authors' contribution

Julia Drewer and Ute Skiba designed the study. Melissa Leduning carried out sample collection with supervision from Julia Drewer, Ute Skiba and Justin Sentian. Julia Drewer, Melissa Leduning, Gemma Purser and James Cash performed analyses with Gemma Purser also advising on data analysis and interpretation. Julia Drewer wrote the first draft of the manuscript and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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