

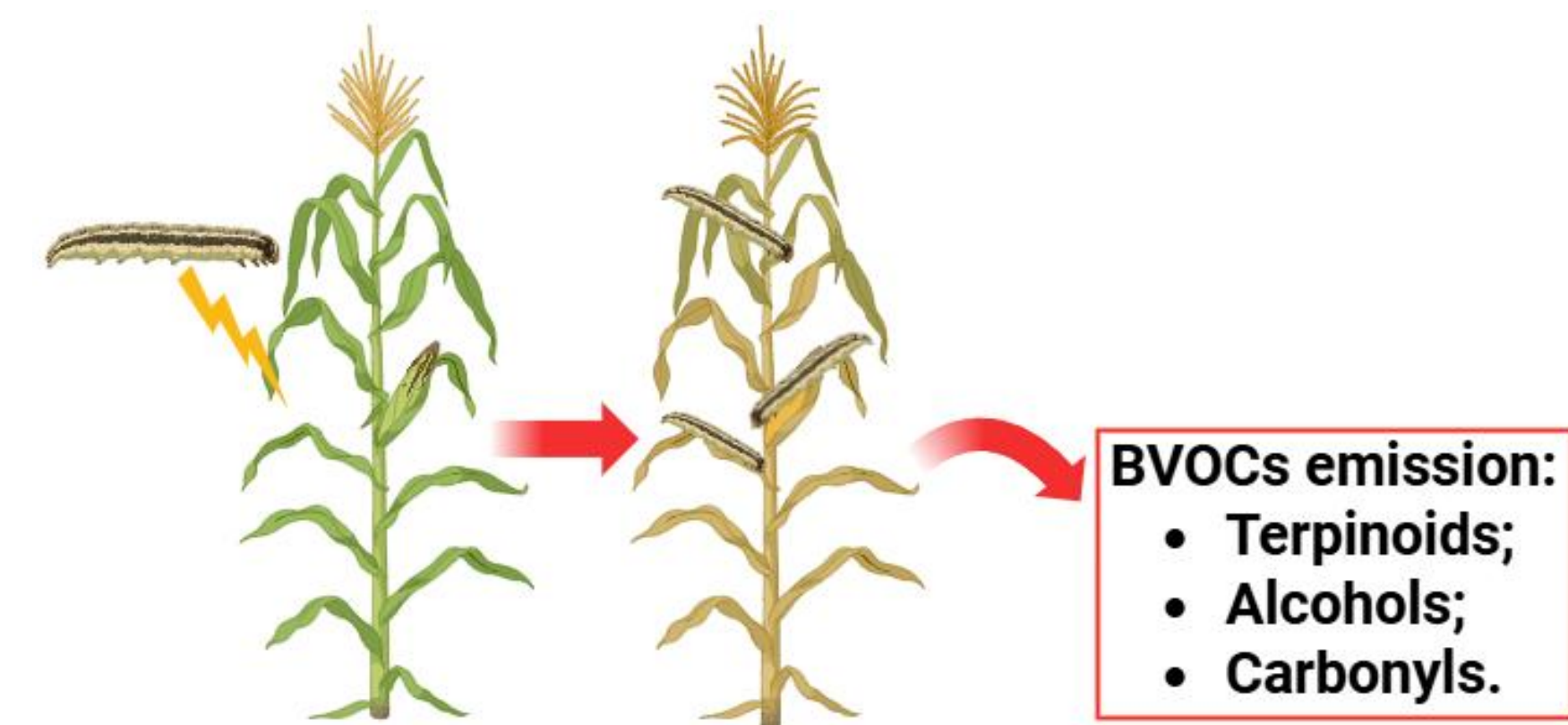
Online monitoring of biogenic volatile organic compounds emitted from Fall Armyworm-infested maize plants with transportable gas chromatography

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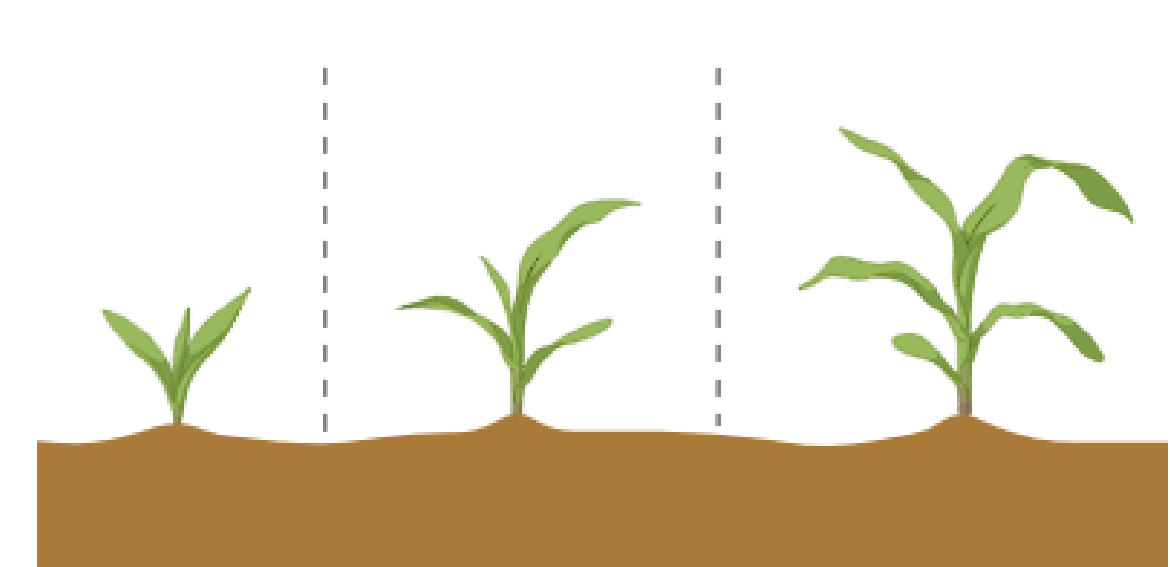
INTRODUCTION

Originating from the tropical regions of America, the **fall armyworm (FAW, *Spodoptera frugiperda*)** is considered an **invasive pest** that could pose a major threat to over **80 plant crops**, particularly **maize plants (*Zea mays*)**¹. In the **European Union (EU)**, FAW infestation is expected to expand after its recent recorded occurrence in **Greece, Portugal, Romania, and Bulgaria**^{2,3}. Some studies reported that infested plants emit **biogenic volatile organic compounds (BVOCs)** that can serve as **tracers of FAW presence**⁴. Reducing the risk of **further spread** involves the development of powerful **analytical tools** that can detect BVOCs at **trace levels** and thus sense pest infestation. Among various methods, **gas chromatography (GC)** stands as a selective and sensitive tool. Coupled with thermal desorption (TD) and lamp photoionization detection (L-PID), it can provide a **continuous, real-time analysis of BVOC signature emissions**.



METHODOLOGY

Maize (*Zea mays*) plant culture

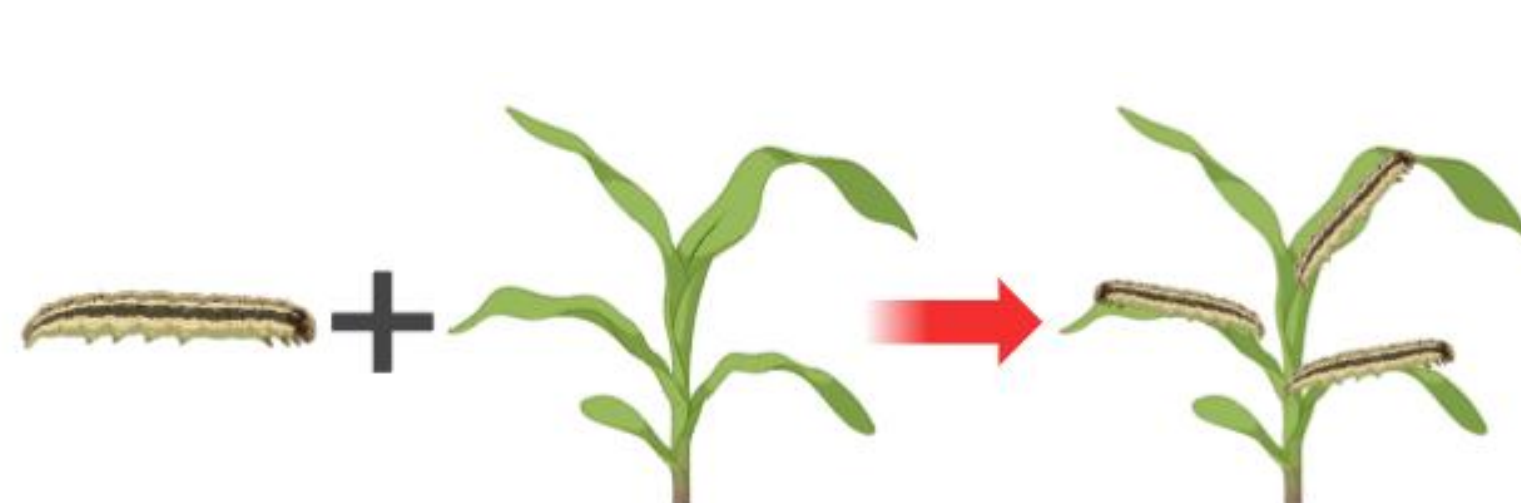


Maize was grown in a greenhouse with control of:

- temperature;
- humidity;
- and light.

The **plant age** for the experiment was chosen to be between 14 and 21 days old.

Infestation with (*Spodoptera frugiperda*)



The **FAW larvae** were grown under strict quarantine conditions.

The **larvae development stage** was chosen to be from early third to late third. The infestation included **2 to 3 larvae** and started a few hours before the start of the **BVOCs measurement**.

The **damage** induced to the plant was quantified in terms of **leaf area**.

Collection of BVOCs



The **infested plant** was enclosed in a **glass bottle** (no ambient air contribution). The device was flushed with **1 L/min of pure air** for **24 h** of measurement⁵.

AirToxic: continuous, online BVOCs' measurement



Working as **TD-GC-L-PID**, this analyzer includes:

- A **heated PTFE sampling line** (100°C);
- A **VOC-specific adsorbent** (Carbopack C/B) to trap and concentrate the VOCs;
- An **MXT-1 analytical column** (30 m × 0.28 mm × 1 μm) to separate VOCs;
- A **lamp photo ionization detector** (10.6 eV);
- A **toluene permeation tube** for calibration.

RESULTS AND DISCUSSION

Recorded BVOCs signature in undamaged versus infested maize plants

- The emitted **BVOC tracers** are recorded to be released in **higher quantities** when the plant is **infested** by the FAW (**Figure 1**, in red) than when the plant is **undamaged** (**Figure 1**, in brown).
- The threshold for the choice of pest infestation tracers was based on the **ratio** value: when the ratio is calculated to be higher than or equal to 2, then the chemical compound is considered to be **specific** for infestation (**Figure 2**).
- The ratio of mean concentration between **infested** and **undamaged** plants is calculated as follows:

$$\text{Ratio} = \frac{\text{Tracer mean toluene - equivalent concentration in infested maize plant}}{\text{Tracer mean toluene - equivalent concentration in undamaged maize plant}}$$

I1 pest-infestation tracer:
retention time **818 seconds**

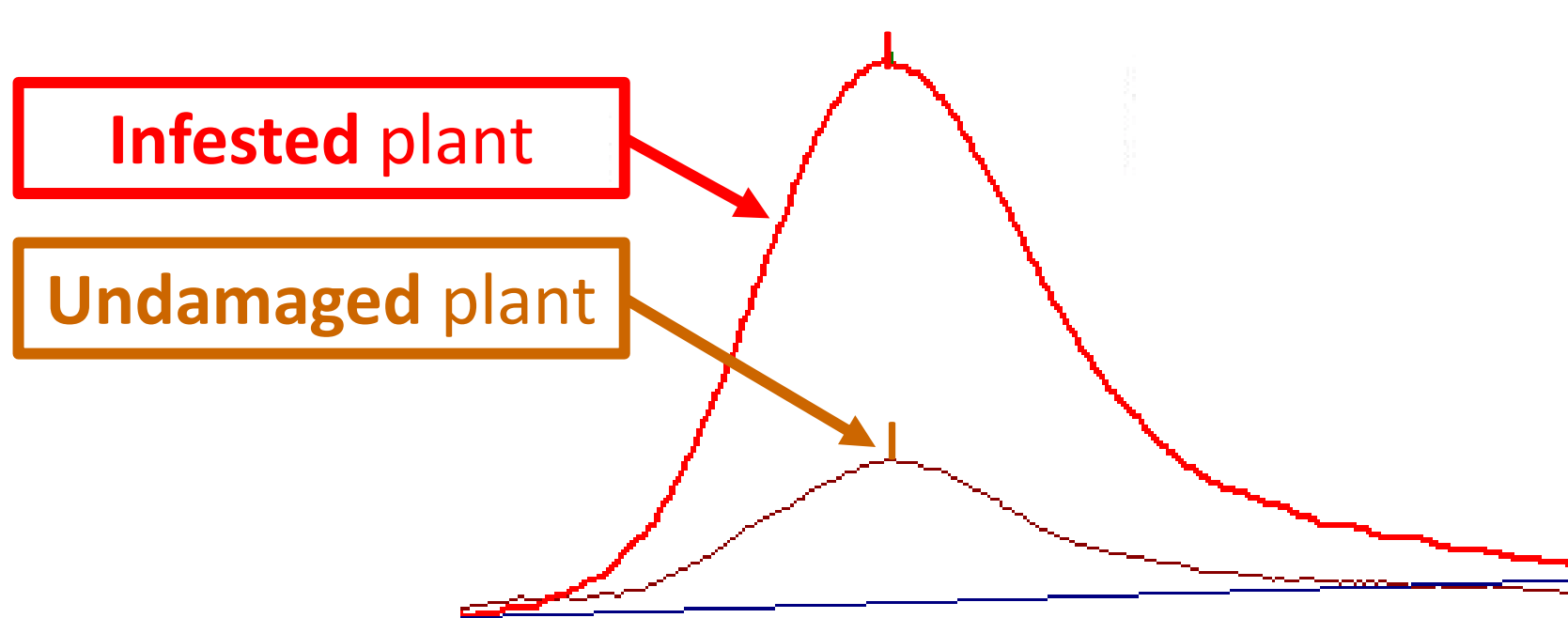


Figure 1: Chromatographic peak of the I1 pest-infestation tracer in the measurements of an infested (top) versus an undamaged (bottom) maize plant.

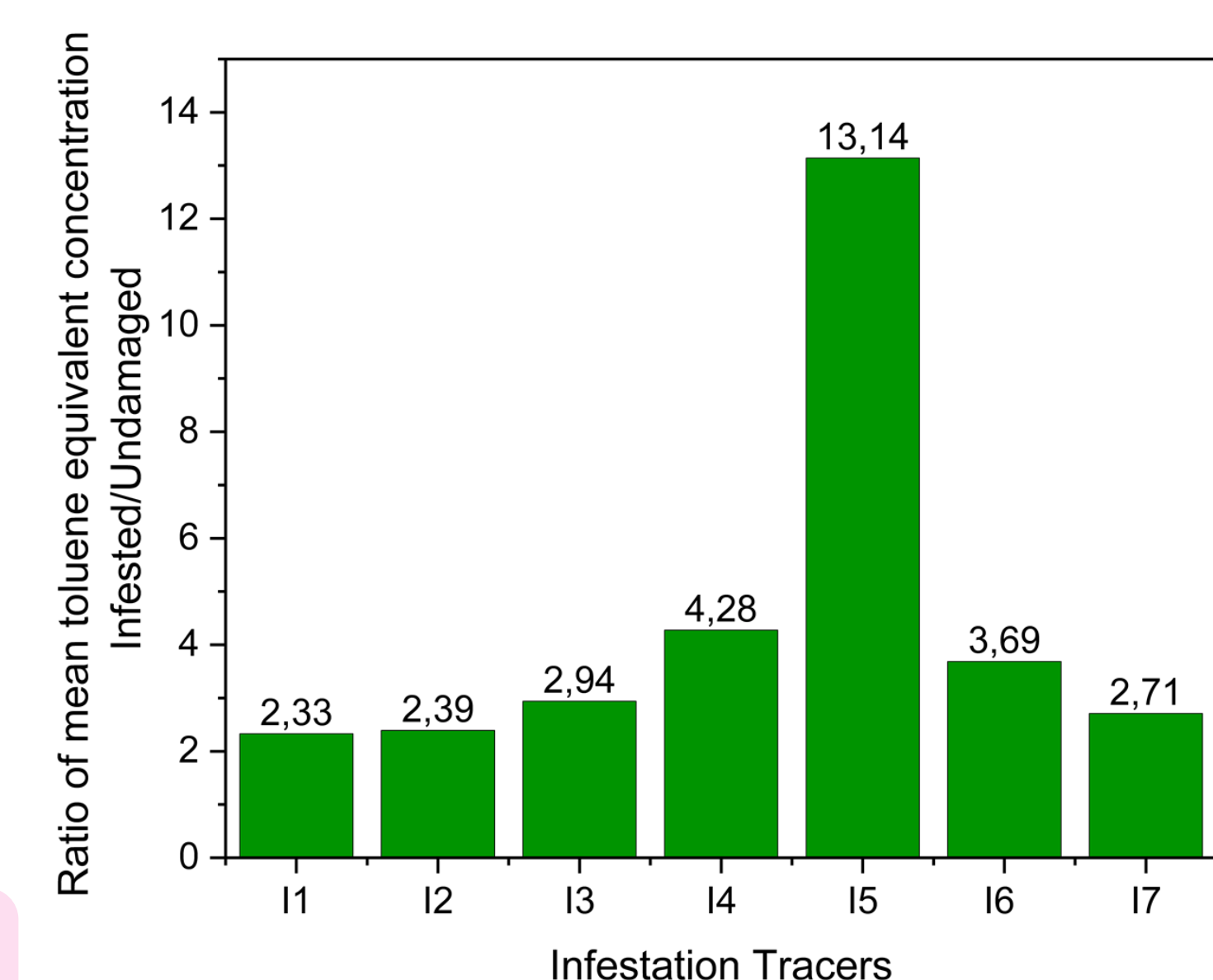


Figure 2: Bar graph of the ratio of mean toluene equivalent concentration between infested and undamaged maize plants.

Temporal evolution of BVOCs concentration

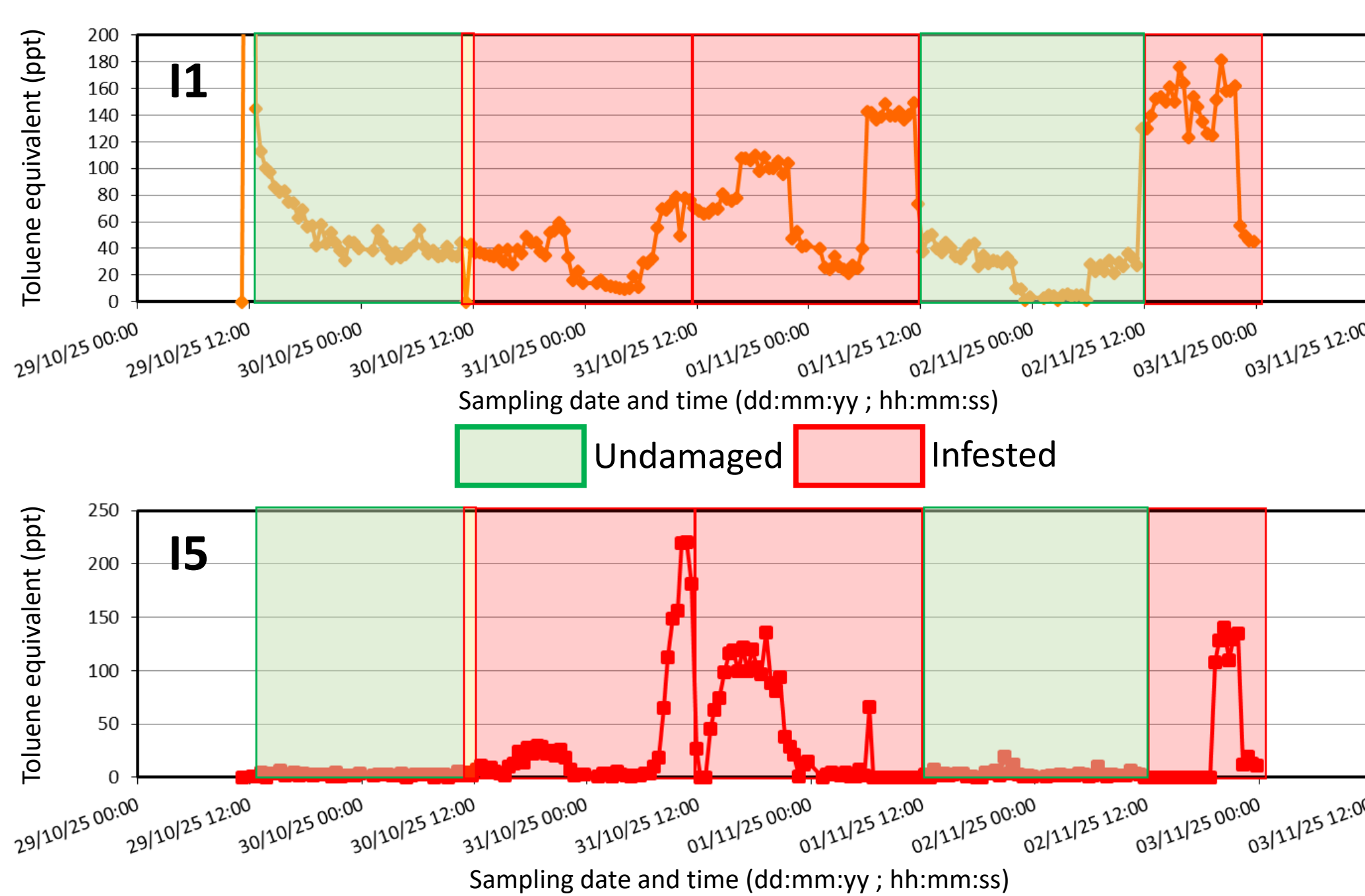


Figure 3: Temporal evolution of pest-infestation tracers I1 (top, orange) and I5 (bottom, red) concentrations over undamaged (green) and infested (red) time slots.

The **temporal trend** of BVOCs emissions was recorded by **24 h** time slot with real-time measurement of **undamaged or infested plants**. This evolution in time highlights the **day and night** difference in emitted tracer concentrations. The recorded BVOC concentrations were in the range **10–2200 ppt**. The tracers quantities emitted were dependent on **several parameters**, such as: the plant age; the number of pest individuals; the voracity of pests (damage).

Statistical analysis: correlation and regression studies

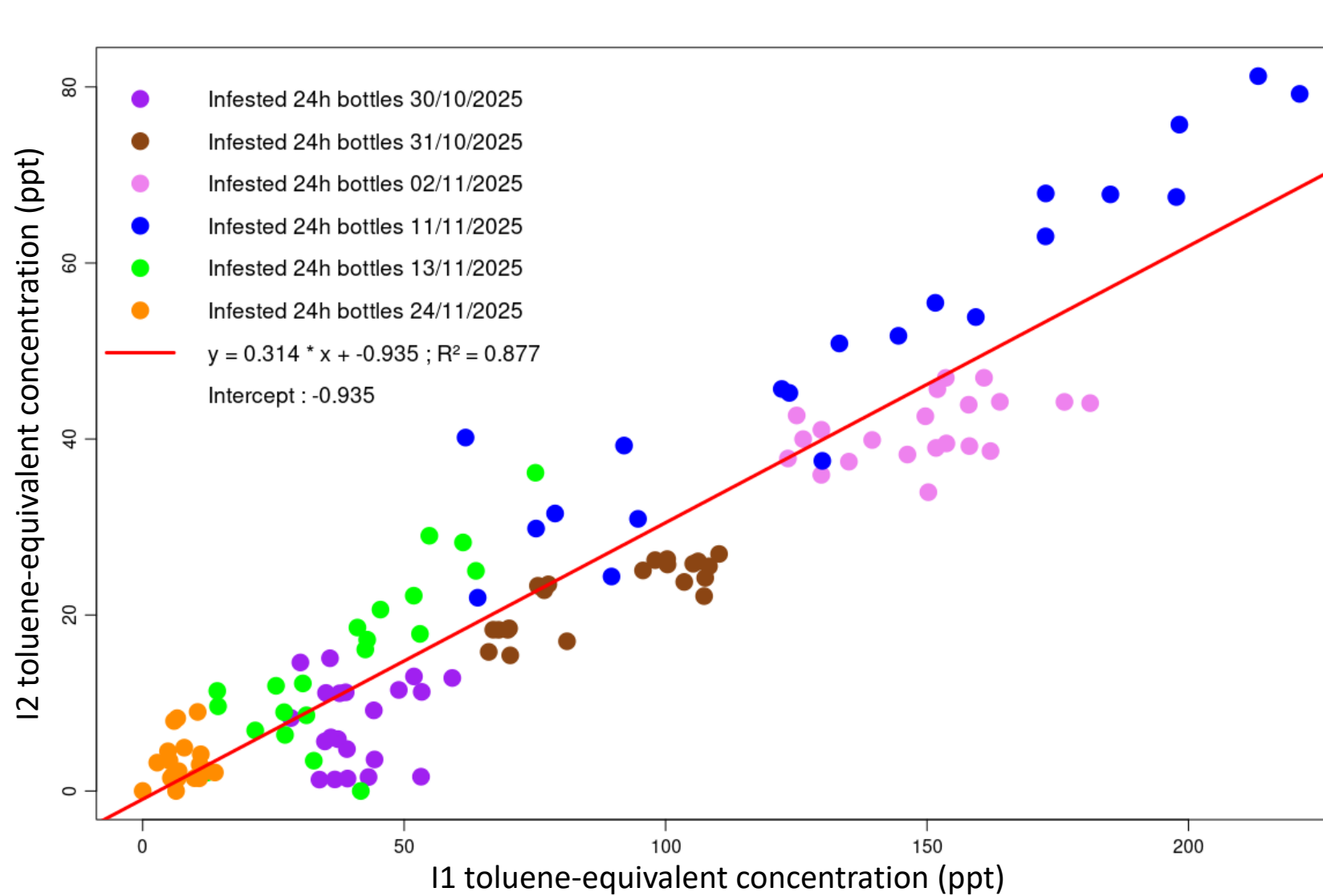


Figure 4: Linear regression for all 24h experiment replicates of infested maize plants for I1 and I2 infestation tracers.

The **regression study** showed a linear correlation between **I1 and I2 tracers** ($R^2 = 0.877$). The statistical analysis was performed taking into account only the infested plants' 24 h-measurements during the **daytime**. Whatever the species, no data recorded during the night were used since a strong decrease was observed when the light was off (**Figure 3**).

Spearman correlation test showed that the evolution between the **I1 and I2 tracer concentrations** was **non-random** (p -value < 0.05).

CONCLUSION AND PERSPECTIVES

The **temporal trend** of BVOC emissions highlighted the day and night evolution of **trace-level** concentrations of BVOCs in the range 10–2200 ppt. The results showed that 24h-continuous measurement **BVOC profiles differ** between **undamaged** and **FAW-infested plants** ($n=12$). **Seven tracers** of the FAW infestation were assigned as **infestation-specific** based on the ratio of mean toluene-equivalent concentrations of BVOCs. **Statistical analysis** demonstrated the same behaviour of some tracers. This field campaign conducted under laboratory-controlled conditions has proven that the online TD-GC-L-PID analyser is **selective and sensitive** (able to detect BVOCs at **trace levels**) demonstrating its capability for **near real-time analysis of BVOC signature emissions**. Further miniaturization of the analyser will enable to obtain a **portable, non-invasive, and non-destructive analytical tool** (μ -TD-GC-L-PID).

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