PRIRODNI REPELENTI: EKSTRAKCIJE I GC-MS ANALIZE HLAPLJIVIH KOMPONENTI KLINČIĆA, LAVANDE I TIMIJANA

NATURAL REPELLENTS: EXTRACTIONS AND GC-MS ANALYSES OF VOLATILE COMPONENTS OF CLOVE, LAVENDER AND THYME

Halyomorpha halys

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Introduction

Volatile organic compounds (VOCs) are low molecular weight chemicals that readily evaporate. In plants, they form the basis of what we recognise as essential oils. Their effects in plants encompass a wide range of biological, ecological, and biochemical processes. Because VOCs readily evaporate and disperse through the air, they serve as a primary medium through which plants communicate with their environment, influence other organisms and defend themselves against threats such as insects [1-2].

In this work, we selected three plants with known repellent activity - clove (flower buds), lavender and thyme (flowering aerial parts) and applied different methods of VOC extraction including hydrodistillation using a Clevenger apparatus (CE), steam distillation (SD) and Soxhlet extraction (SE). The aim is to compare the volatile profiles of extracts with potential repellent activity. The extracts obtained are analysed by proper GC-MS and HS-SPME techniques. The results will provide a better understanding of the influence of the extraction method on the chemical composition of the extracts and their potential for use as natural repellent agents.

Extraction methods

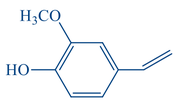
Clevenger: Extraction is carried out using 20 g of plant material and 250 mL of distilled water, with heating for 2 h. The essential oil (**EO**) (Figure 1) and hydrodistillate (**HD hydrolat**) are then obtained.

Steam distillation: The extraction is carried out with 20 g of plant material, and 200 mL of hydrolate is collected. The hydrolate (100 mL) is extracted by liquid–liquid extraction with *n*-hexane and the solvent is evaporated. This yields the hydrolate (**SD hydrolat**) and the evaporated extract (**SD extract**).

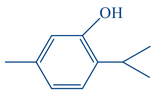
Soxhlet extraction: Extraction is carried out with 5 g of plant material in 50 mL of *n*-hexane. After 2 h, the resulting solution is filtered and the solvent is evaporated (**Soxhlet**).

The volatile profiles of essential oils were analysed by **GC-MS**, and those of hydrolates and solid extracts by **HS-SPME GC-MS**.

Essential oil of clove (**CL_EO**)

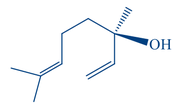


Eugenol



Thymol

Essential oil of thyme **THY_EO**

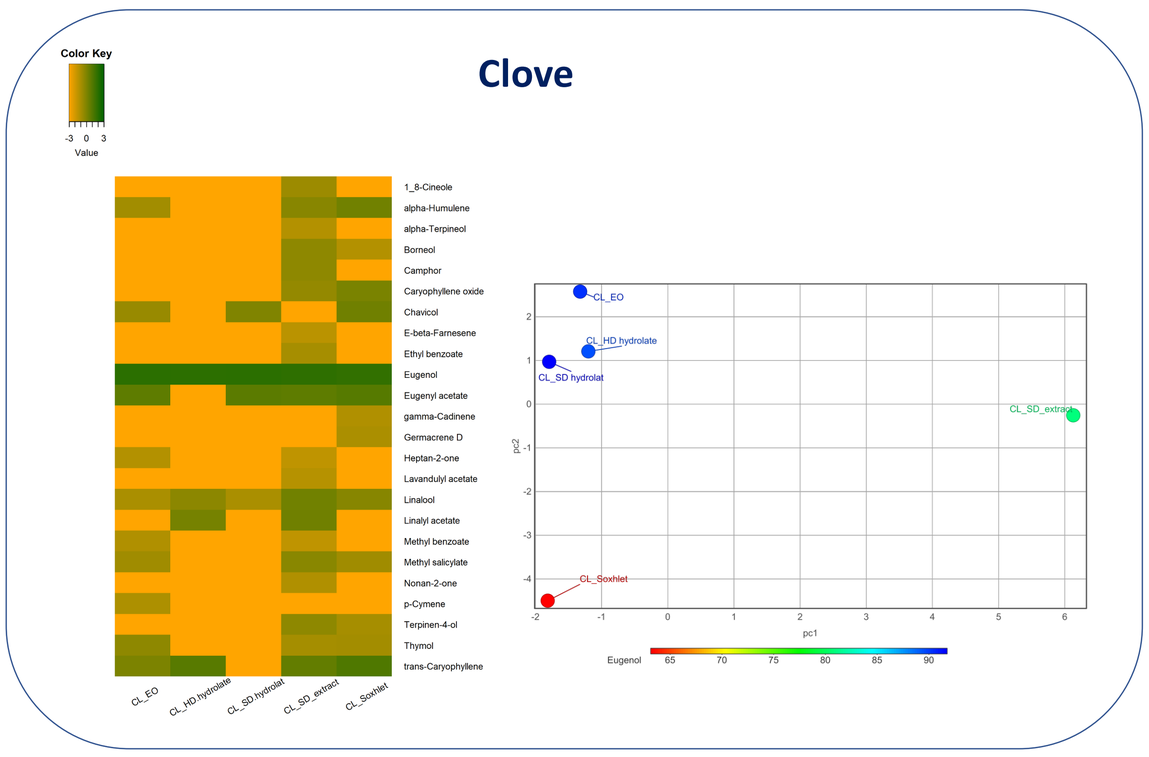


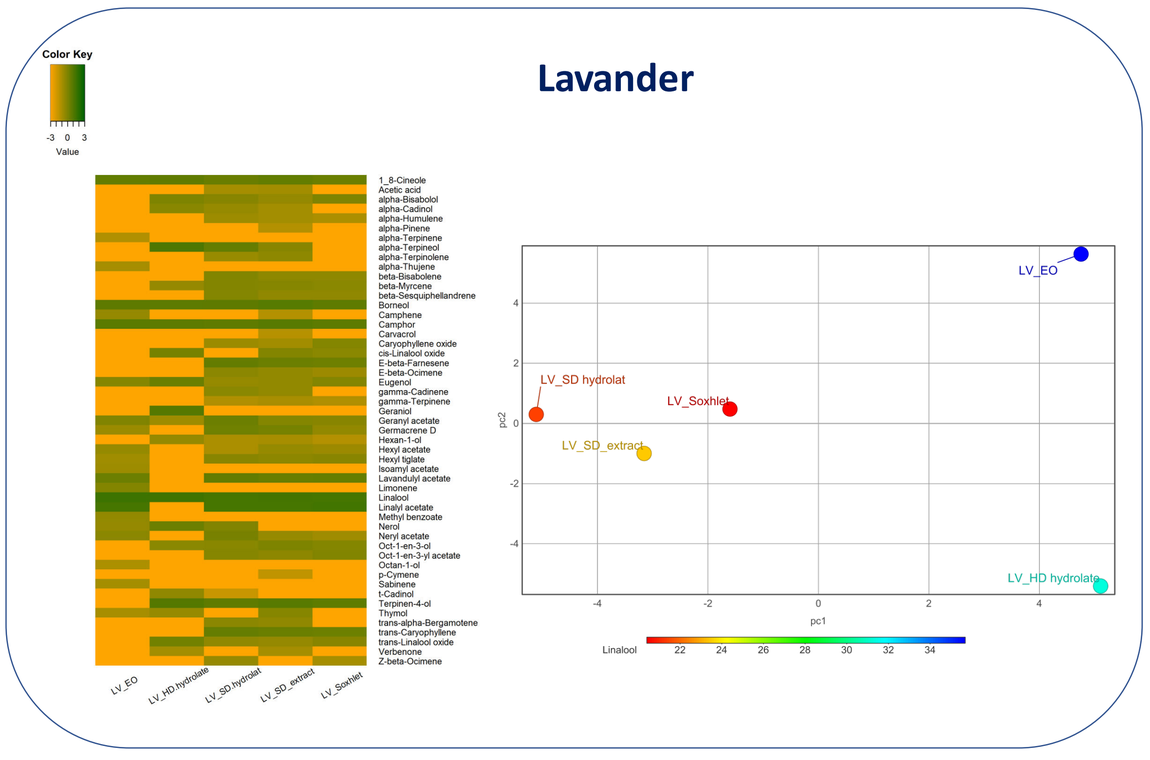
(*R*)-(-)-Linalool

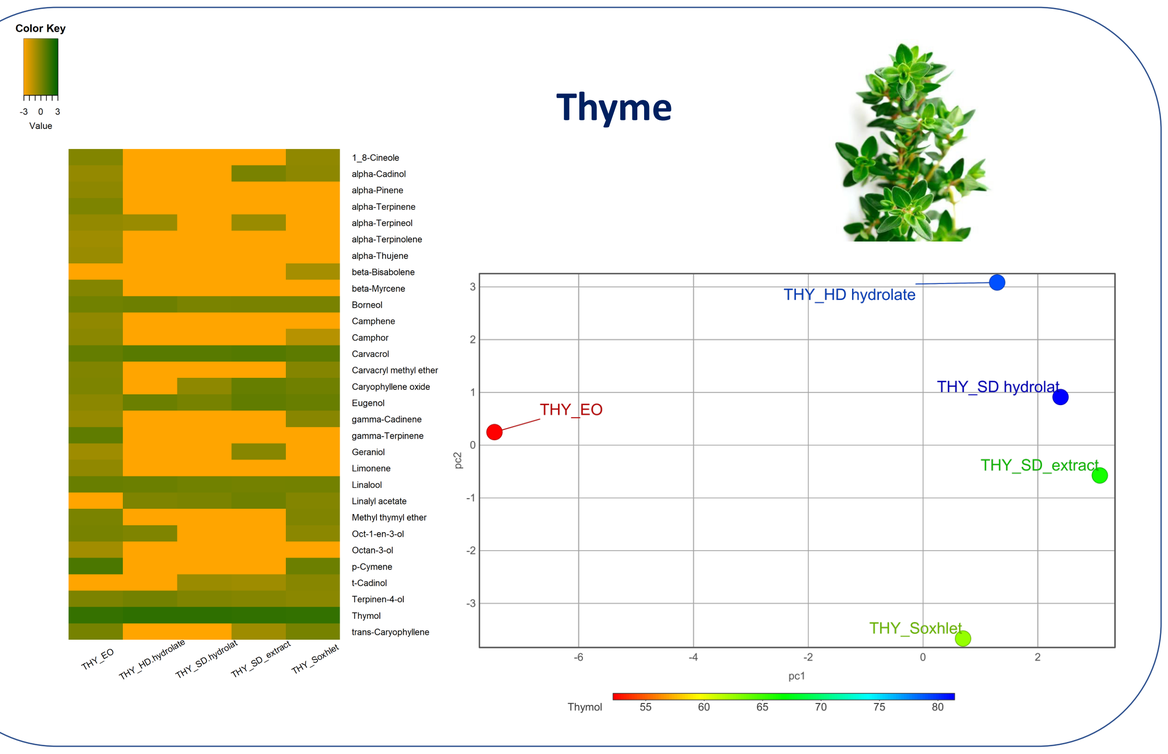
Essential oil of lavender (**LV_EO**)

Figure 1. Essential oils produced and analysed in this study and their major constituents.

Figure 2. Heatmaps showing the volatile profiles of clove, lavender, and thyme essential oils as determined by GC–MS, and of the corresponding hydrolates and solid extracts as determined by HS-SPME. The heatmaps were generated using the heatmap.2() function in R, with data expressed as log10-transformed chromatographic peak area percentages (%). The corresponding principal component analysis (PCA) score plots, generated using DataWarrior, illustrate the clustering of extracts with similar volatile profiles and the separation of those with distinct profiles.







Literature

[1] Md. H. Bin Nabi, Md. M. Ahmed, et al., *Food Chemistry Advances* **8** (2025) 101048.

[2] R. Ayllón-Gutiérrez, L. Díaz-Rubio, et al, *Agriculture* **14** (2024) 1766.

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Conclusion

- Essential oils were obtained by extraction using a Clevenger apparatus, and the yield was expressed relative to the plant material. The highest yield was obtained from clove (1.15 %), followed by lavender (0.625 %), with the lowest from thyme (0.165 %).
- Using the other two extraction methods (SD and SE), the yield depended on both the plant material and the method. The highest yield was again obtained when clove was used as the plant material (SD: 4.05 %; SE: 3.85 %), followed by lavender (SD: 1.75 %; SE: 3.56 %) and thyme (SD: 0.66 %; SE: 2.00 %).
- All extracts obtained, including hydrodistillates, were analysed for their volatile components using GC-MS and HS-SPME techniques. It can be concluded that the composition of essential oils, as well as other extracts, varies depending on the plant material used.
- All extracts from a given plant contained the same components, but in different proportions.
- The dominant components of the same plant were found in all extracts, regardless of the extraction method, but in different proportions. In clove, eugenol is present in all extracts, with the highest proportions of 91.78 % in the hydrolate and 90.48 % in the essential oil. The dominant compounds in lavender are linalool and linalyl acetate. In all lavender extracts, linalool is present, with a proportion of 35.69 % in the essential oil and the lowest proportion, 20.4 %, in the extract obtained by SE. Linalyl acetate was obtained in the highest proportion in the SE extract (27.21 %), while it was not detected in the hydrodistillate. The component with the highest proportion in thyme is thymol, which is present in all extracts, with the highest proportion in the hydrolate (81.44 %), and the lowest proportion in the essential oil (52.2 %). Thyme essential oil, unlike the other thyme extracts, has another dominant component, *p*-cymene, with a proportion of 16.43 %.

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