

Determination of pyrrolizidine alkaloids in oregano by a miniaturized QuEChERS method combined with UHPLC-IT-MS/MS

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Gold statement

- Miniaturized QuEChERS method with Green Analytical Chemistry requirements
- Chromatographic separation of 21 pyrrolizidine alkaloids within 10.5 min by ultra-high liquid chromatography coupled to ion-trap tandem mass spectrometry
- Analysis of 21 pyrrolizidine alkaloids in 23 oregano samples to ensure food safety

Introduction

In the last two years it has been striking the significant increase in the number of food alerts reported on the Food and Feed Safety Alerts (RASFF) portal related to the presence of pyrrolizidine alkaloids (PAs) and their oxidized forms (pyrrolizidine alkaloids N-oxides, PANOS) in different foodstuffs [1], being oregano the product with the highest incidence. PAs are natural plant toxins generated as a defence mechanism against herbivores and insects. Their intake is mainly associated with liver damage, but some of them have also been considered genotoxic and carcinogenic (Category 2B) by the International Agency for Research on Cancer [2].

Body

To broad knowledge about the occurrence of these contaminants in aromatic herbs and ensure their food safety, the aim of this work was to develop a sustainable analytical methodology by ultra-high liquid chromatography coupled to ion-trap tandem mass spectrometry (UHPLC-IT-MS/MS) to monitor the presence of the 21 PAs/PANOs currently suggested by the European Food Safety Authority (EFSA) in oregano samples. Accordingly, the multicomponent extraction of the target analytes from the matrix was achieved by the miniaturization of the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) procedure by reducing in ten times the amounts of sample, solvents and partitioning salts employed, leading to an improved cost-effective and environmentally friendly microextraction method, which meets the Green Analytical Chemistry principles.

The chromatographic separation was achieved within 10.5 min and the analytical method was successfully validated (recoveries from 77-96% and relative standard deviations <13% (n=9)) and applied to the analysis of 23 oregano samples. It was revealed that all the samples analysed were contaminated with PAs and PANOs. Moreover, in a 30% of them the sum of the total PAs/PANOs exceeded from 1000 µg/kg, which is the maximum concentration allowed for these compounds currently under discussion by the EU Commission for aromatic herbs.

Conclusion

This work confirms oregano is highly contaminated with concerning values of PAs/PANOs that may entail potential health risks for consumers. Therefore, this highlights the great need to establish an official regulation with maximum concentration levels to monitor the occurrence of these compounds, so that food safety can be ensured.

References

[1] RASFF - Food and Feed Safety Alerts, available online: <https://webgate.ec.europa.eu/rasff-window/portal/?event=SearchForm&cleanSearch=1> (accessed on 13 July 2020)

[2] B. Dusemund, *Food Chem. Toxicol.* **115** (2018) 63-72.