

Two-step green extraction (SFE-CO₂ + ASE) produces low-contaminant extracts from olive mill wastewater sludge for functional applications



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INTRODUCTION & AIM



Olive mill wastewater (OMW) sludge
Environmental concern
Opportunity for valorization

Green extraction technologies
Supercritical CO₂ extraction (SFE)
Accelerated solvent extraction (ASE)

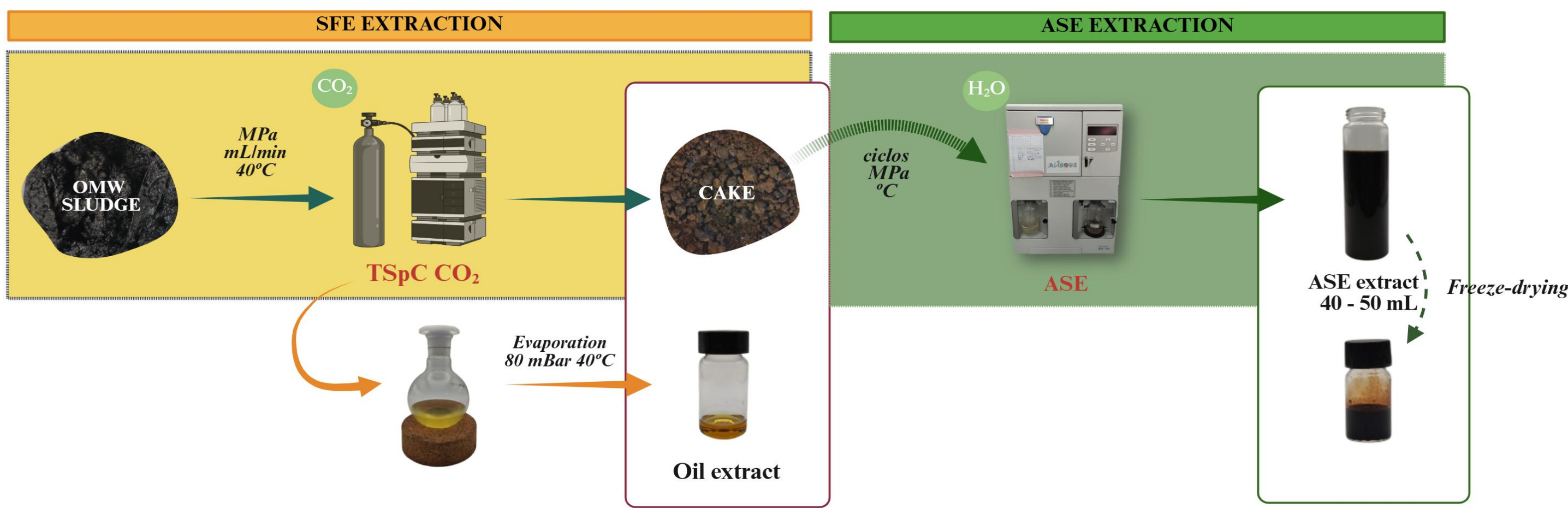
CHALLENGE
Extraction methods that recover bioactives while minimizing metal and mycotoxins transfer

The aim is to evaluate how the **SFE → ASE** sequence affects the **distribution** and transfer of heavy metals (As, Cd, Hg and Pb) and mycotoxins in OMW sludge in order to obtain **safe extracts** suitable for valorization.

- ❖ Quantify the contaminants (heavy metal and mycotoxins) in fresh OMW sludge from six ponds (TED-13, 14, 15, 16, 20, 22).
- ❖ Use **mass balance** to determine metal partitioning across the SFE-ASE workflow, assessing retention in cakes and transfer to SFE oils and ASE aqueous fractions.
- ❖ Promote sustainable waste valorization by obtaining **bioactive-rich extracts** with low contaminant levels.

METHOD

SEQUENTIAL EXTRACTION FOR RECOVERY OF LIPIDS AND POLYPHENOLS



ICP-MS EVALUATION OF HEAVY METAL CONTENTS

FRESH OMW SLUDGE
SFE CAKE
EXTRACTS



RESULTS & DISCUSSION

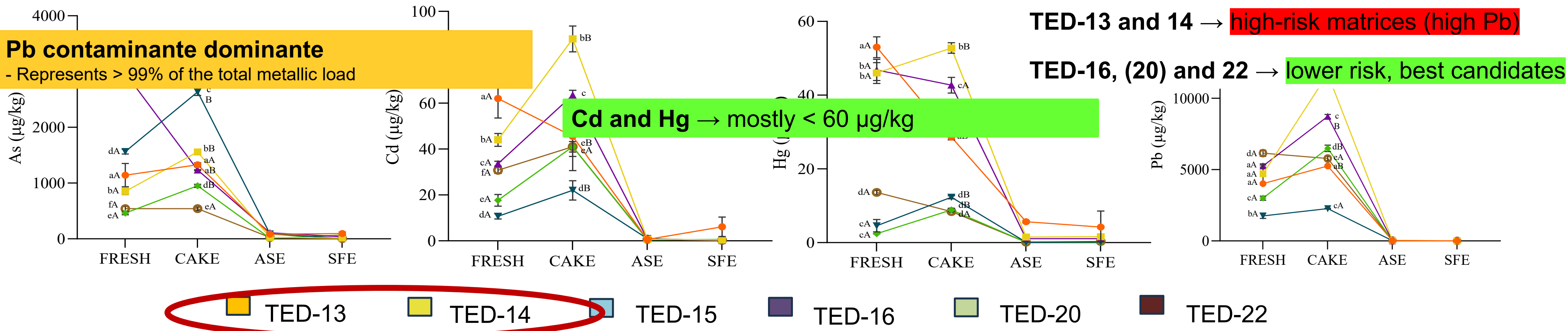


Figure 1. Heavy metal content in fresh olive mill wastewater (OMW), SFE cake, and extracts obtained by SFE and ASE methods. The results were expressed in µg/kg. (p < 0.05) according to Tukey's test.

Compliance with EU 2023/915: ~ 26/30 extracts are below 5% of the limit, with the highest (TED-13/14) at less than 10%

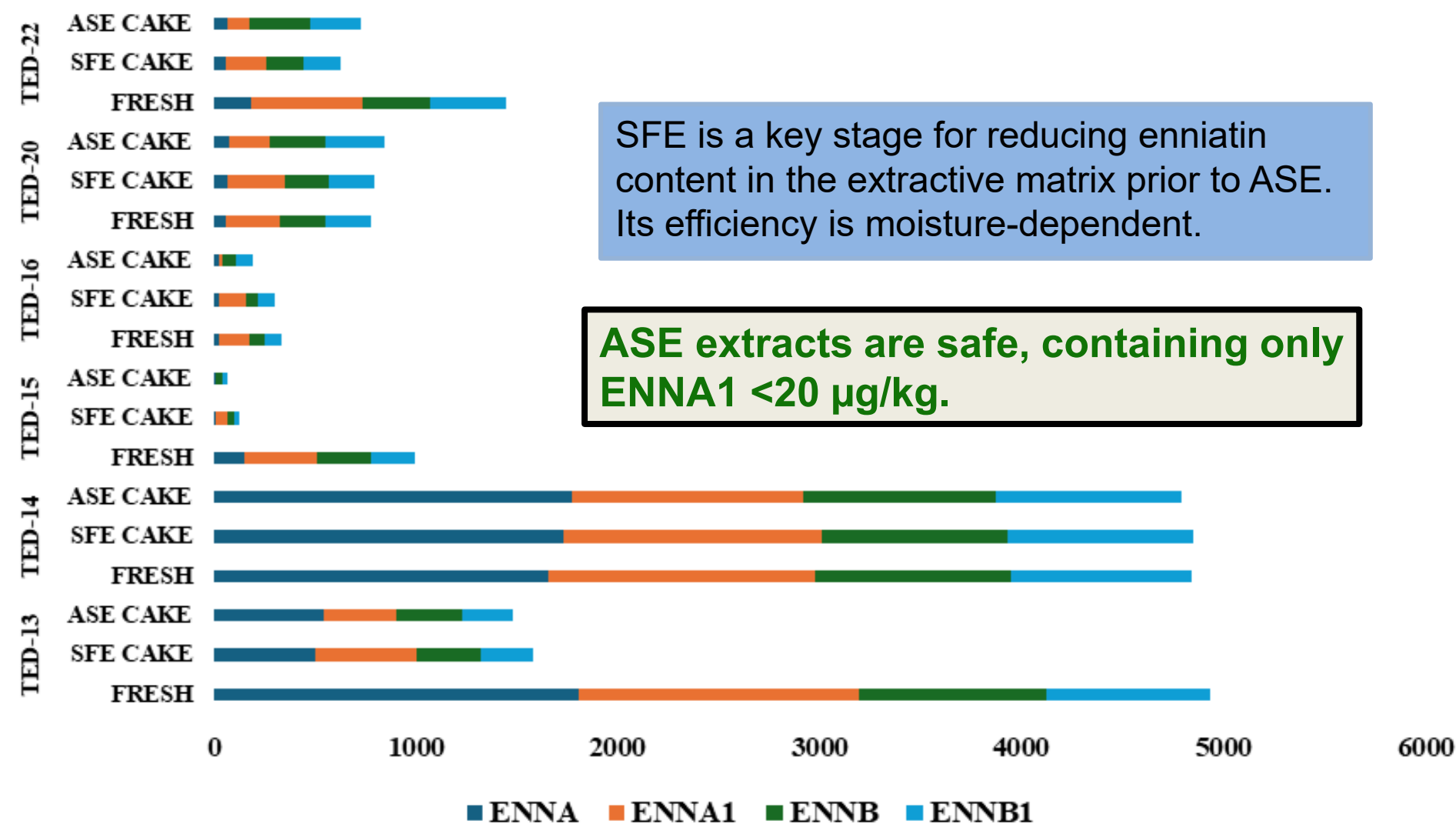


Figure 2. Concentration of enniatins (ENNA, ENNA1, ENNB, and ENNB1) in fresh olive mill wastewater (OMW) sludge, SFE cake, and ASE cake from TED ponds.

CONCLUSION

- ✓ **Pb dominates in fresh sludge and varies widely across ponds; TED-16 and TED-22 are best for valorization.**
- ✓ **SFE does not remove heavy metals, but it can extract up to 70% of enniatins.**
- ✓ **ASE may mobilize some residual metals, but final extracts stay far below EU 2023/915 (~26/30 <5% of limits; worst cases <10%).**
- ✓ **ASE extracts contained only trace levels of ENNA1 (<20 µg/kg), consistent with a toxicologically safe profile**
- ✓ **SFE + ASE process produces oil and aqueous extracts whose heavy metal and enniatin concentrations remain below current food safety limits**

REFERENCES

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Jestoi, M. Emerging Fusarium -Mycotoxins Fusaproliferin, Beauvericin, Enniatins, And Moniliformin.A Review. *Crit. Rev. Food Sci. Nutr.* 2008, 48, 21–49, doi:10.1080/10408390601062021.