

LIFE CYCLOPS: Upcycling polyphenols from industrial olive oil waste

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Keywords: Agrifood Byproduct Revalorization, Polyphenols, Ultrafiltration, Nanofiltration

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Olive oil production plays a pivotal role in the economy in Mediterranean countries, being one of the most important industrial activities. However, it results in a substantial byproduct known as alperujo, consisting of a mixture of water, olive solids, and fatty residues. Spain, the largest producer of olive oil, yielded approximately 1.2 million tons in the 2019/2020 campaign, followed by Italy and Greece with outputs of 0.34 and 0.3 million tons respectively. Consequently, these three countries collectively generate over 7 million tons of alperujo annually. Improper disposal poses legal and environmental challenges due to its polluting nature, leading to soil degradation in nutrient content, pH alteration, increased conductivity, reduced water retention capacity, odour issues, and potential pathogen transmission (Garcia-Ortiz, C, 2016). Moreover, due to its recalcitrant nature, the presence of polyphenols in wastewater hinders the application of traditional biological treatments (Muñoz-Palazon, B., 2023).

To tackle this concern, whereas seizing a novel business opportunity, this study explores the revalorization of this agricultural waste within a circular economy framework by focusing on the extraction and purification of polyphenolic compounds from wet olive pomace using a sustainable approach. The recovered polyphenols will become high added-value products for alimentary, cosmetics or pharmaceutical industries, among others. Moreover, the de-phenolized waste will be sent to an anaerobic digestion stage to produce biogas, thus promoting a zero waste achievement.

The extraction method employed in this work involved solid-liquid extraction using water as the solvent, mirroring a green liquid extraction technique, since water can be considered as an environmentally friendly solvent. The extraction process occurred in 10L tanks at ambient temperature for 30 min with constant stirring without altering the pH of the sample. The final conditions were optimised through an experimental design, considering factors such as temperature, time, pH, and the ratio of sample to water (w/v). The total polyphenolic content (TPC) was quantified by the Folin-Ciocalteu method and polyphenols were identified and quantified by HPLC-HRMS/MS and HPLC-UV, respectively.

After extraction, to achieve the purification of the polyphenolic fraction, a sequential membrane process was studied. It involved a ceramic ultrafiltration (UF) step followed by a nanofiltration (NF) step, with different objectives. On the one hand, the UF effectively eliminated suspended solids and major molecular weight compounds, thereby clarifying the extract. On the other hand, the NF concentrated the target compounds, contributing to the substantial enhancement of the TPC of the extract.

This study conducted a comprehensive analysis of the extracted compounds, revealing a diverse range of antioxidant both phenolic and non-phenolic constituents (Figure 1, Table 1). Purification and concentration of polyphenols has been conducted in a membrane train based on two steps. Ultrafiltration rejected colloidal compounds and diminished COD, thus indicating a more purified extract while Nanofiltration achieved polyphenol concentration. Polyphenols have been concentrated from 674 mg/L at the entrance of the ultrafiltration membrane to 1929 mg/L after passing through the nanofiltration membrane (Figure 2). Additionally, the performance of the proposed technology was further assessed by the quantification of two key compounds, all of which hold significant potential for various applications: hydroxytyrosol glucoside and p-coumaric acid glucoside, with concentrations of 117, and 38 (ppm) in the nanofiltration concentrate, and rejections of 98.5% and 98.9%, respectively.

This environmentally friendly approach not only demonstrates the feasibility of polyphenol extraction from wet olive pomace but also underscores the potential of revalorizing agrifood residues. The sustainable reuse of this waste stream aligns with the principles of a circular economy, where resources are efficiently managed, and waste materials are transformed into valuable products. Furthermore, this work highlights the promise of transitioning from laboratory-scale processes to a pilot plant level, emphasising the potential of wet olive pomace as a valuable resource for the recovery of phenolic compounds. In conclusion, the technology developed in this work could substitute the current process for polyphenol extraction from virgin raw materials.

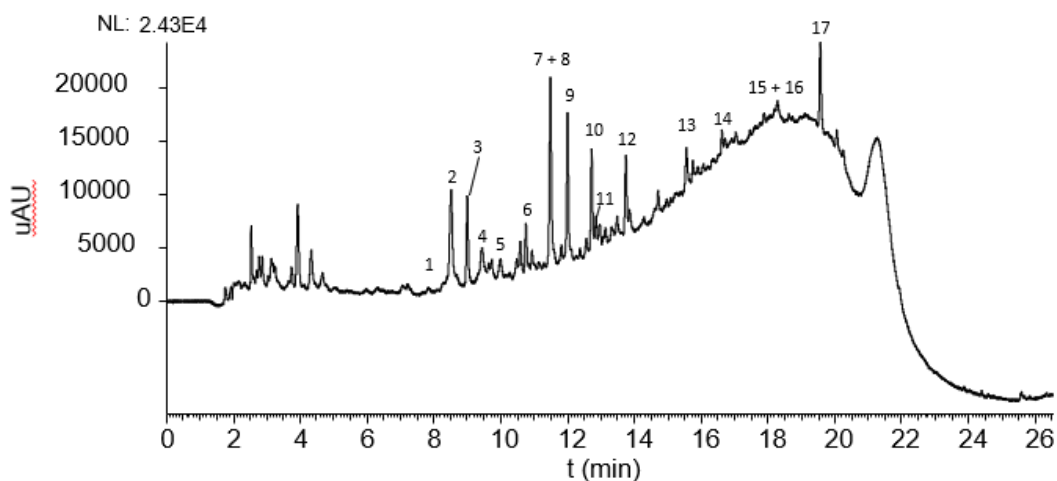


Figure 1. Comparison between theoretical predictions and experimental measurements.

Table 1. Comparison between theoretical predictions and experimental measurements.

Peak	Compound	Peak	Compound
1	3-Hydroxytyrosol	10	EA glucoside
2	Hydroxytyrosol Glucoside	11	Decarboxymethylated elenolic acid (EDA)
3	Loganic acid	12	EA derivative
4	Loganic Derivative	13	p-coumaric acid
5	Hydroxy-EDA	14	Elenolic acid
6	Hydroxy-EA	15	Caffeoyl-Secologanoside
7	p-coumaric acid glucoside	16	Apigenin rutinoside
8	Secologanoside	17	Comselogoside
9	Loganic acid 7-O-acetyl		

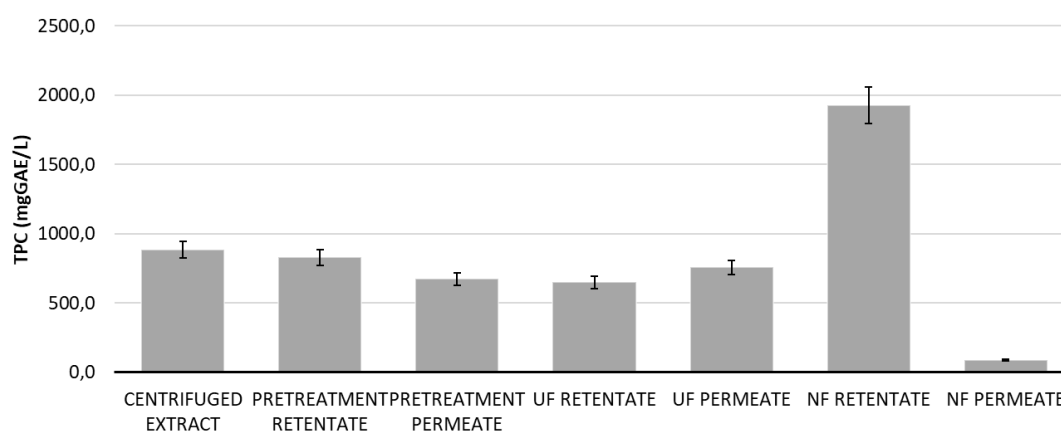


Figure 2. Comparison between theoretical predictions and experimental measurements.

References

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Acknowledgements

This work has been funded by the European Union through the project LIFE CYCLOPS (LIFE21-ENV-ES-CYCLOPS 101074544)