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Microwave hydrodiffusion and gravity: An emergent technology for green extraction of non-volatile compounds

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1. Introduction

Microwave technologies are more and more present in food applications due to their performance in shortening the time of treatments such as drying, pasteurization, defrosting, or post-harvesting [1]. While solvent-free microwave extraction has been extensively used as a green procedure for essential oil and volatile compounds from aromatic herbs [2], its applications have been extended to enhance extraction of phytocompounds simultaneously with drying. In microwave drying, operational cost is lower because energy is not consumed in heating the walls of the apparatus or the environment [3].

2. Objective

- Evaluation of different plant material drying process using a microwave hydrodiffusion and gravity laboratory microwave oven (NEOS-GR, Milestone, Italy) while evaluating the possible co-extraction of non-volatile compounds such as: free sugars, fibers, colour and phenolic compounds.

3. Material and Methods

I. Plant Material

i) Brocolli By-products (90% humidity):

"Florets" "Stalks"

ii) Apple pomace (86% humidity):

iii) Spent Coffee Grounds (65% humidity):

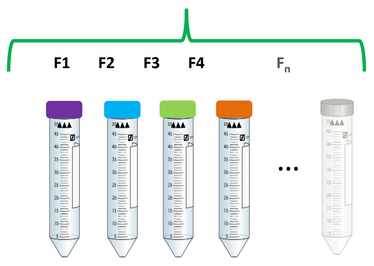
iv) *Pterospartum tridentatum* inflorescences (re-hydrated):

v) Brown Algae (re-hydrated):

II. NEOS-GR experiments

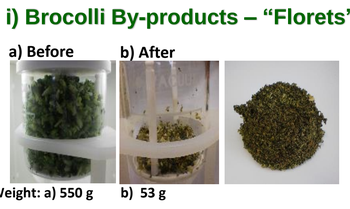
III. Analyses

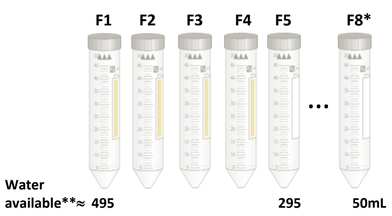
- ❑ Mass yield
- ❑ Absorbance
- ❑ Sugars
- ❑ Phenolics
- ❑ Caffeine



4. Results

i) Brocolli By-products – "Florets"





*Burning point. Complete dryness of plant material.
** Initial water available inside the plant matrix.

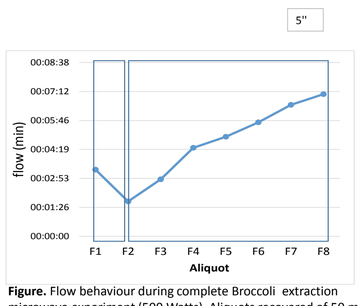


Figure. Flow behaviour during complete Broccoli extraction microwave experiment (500 Watts). Aliquots recovered of 50 mL.

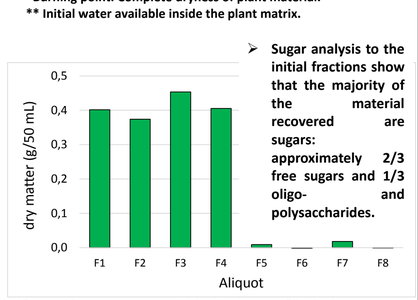
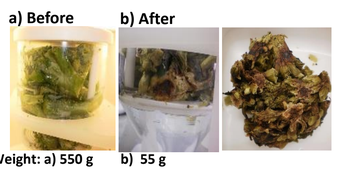


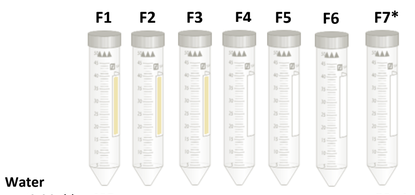
Figure. Mass recovery and distribution through 50 mL aliquots during complete microwave extraction (until achieving burning point at total dryness of the matrix).

➢ **Sugar analysis to the initial fractions show that the majority of the material recovered are sugars: approximately 2/3 free sugars and 1/3 oligo- and polysaccharides.**

- ❖ Six **different broccoli by-products** were tested. In all cases, flow behavior was similar: after the initial heating time, **flow was constant** until achieving matrix burning point (total dryness).
- ❖ **Brocolli Florets** had shrink cylinder shape at the end of the extraction. The same did not happen with **Stalks**.
- ❖ Good recoveries of material were observed during the **initial four extraction cycles**. The remaining period of time was only used to achieve total dryness.
- ❖ Initial fractions were considered for further phytochemicals identification and characterization.

i) Brocolli By-products – "Stalks"





*Burning point. Complete dryness of plant material.
** Initial water available inside the plant matrix.

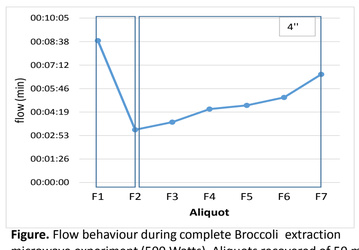


Figure. Flow behaviour during complete Broccoli extraction microwave experiment (500 Watts). Aliquots recovered of 50 mL.

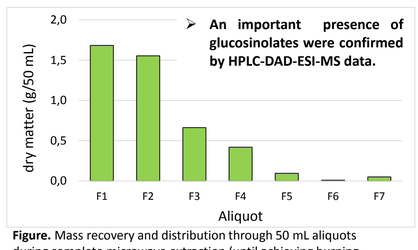
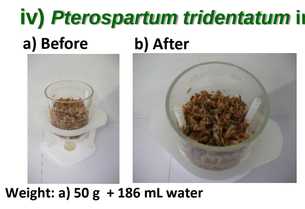


Figure. Mass recovery and distribution through 50 mL aliquots during complete microwave extraction (until achieving burning point at total dryness of the matrix).

➢ **An important presence of glucosinolates were confirmed by HPLC-DAD-ESI-MS data.**

iv) *Pterospartum tridentatum* inflorescences



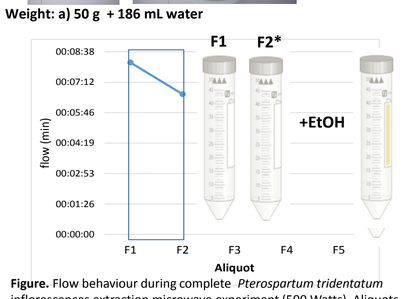
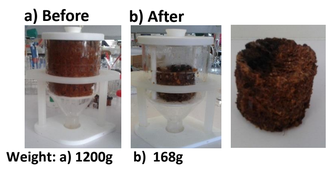
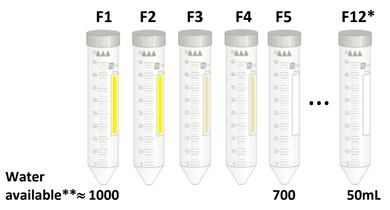


Figure. Flow behaviour during complete *Pterospartum tridentatum* inflorescences extraction microwave experiment (500 Watts). Aliquots recovered of 50 mL.

- ❖ A very fast drying process occurred. Although, approximately 200 mL of water were added, burning point (after complete dryness of plant material) was achieved already at the second fraction recovered.
- ❖ As re-hydration was necessary for microwave application, it seems that the process was not efficient for recovery of material from low water content matrices such as *Pterospartum tridentatum* inflorescences.
- ❖ When using water:ethanol (50:50) co-addition a yellow color extract is recovered.
- ❖ Contrary to Broccoli or apple pomace matrices, no changes were observed in the sample morphology during extraction process.

ii) Apple pomace





*Burning point. Complete dryness of plant material.
** Initial water available inside the plant matrix

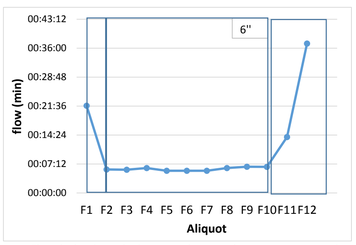


Figure. Flow behaviour during complete apple pomace extraction microwave experiment (500 Watts). Aliquots recovered of 50 mL.

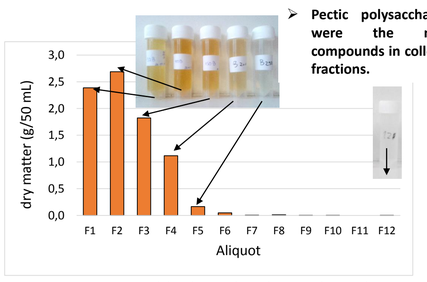
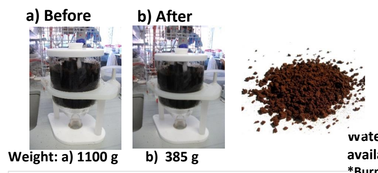
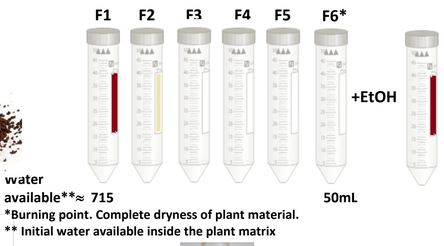


Figure. Mass recovery and distribution through 50 mL aliquots during complete microwave extraction (until achieving burning point at total dryness of the matrix).

➢ **Pectic polysaccharides were the major compounds in collected fractions.**

iii) Spent Coffee Grounds





*Burning point. Complete dryness of plant material.
** Initial water available inside the plant matrix

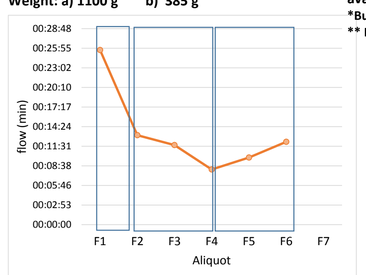


Figure. Flow behaviour during complete spent coffee grounds extraction microwave experiment (400 Watts). Aliquots recovered of 50 mL.

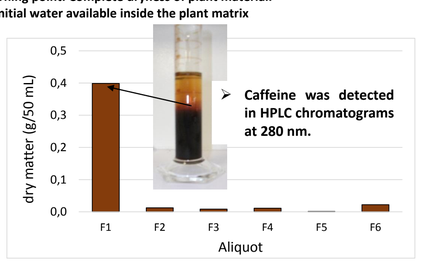
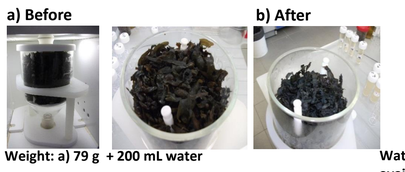
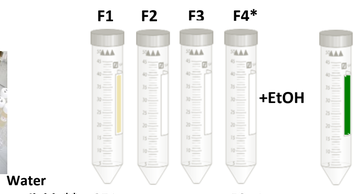


Figure. Mass recovery and distribution through 50 mL aliquots during complete microwave extraction (until achieving burning point at total dryness of the matrix).

➢ **Caffeine was detected in HPLC chromatograms at 280 nm.**

v) Brown Algae





*Burning point. Complete dryness of plant material.
** Initial water available inside the plant matrix

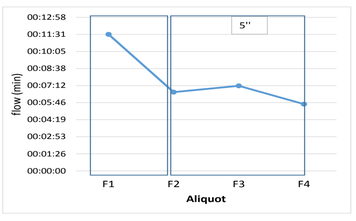


Figure. Flow behaviour during complete brown algae extraction microwave experiment (400 Watts). Aliquots recovered of 50 mL.

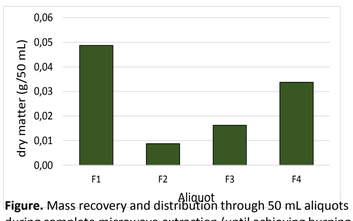


Figure. Mass recovery and distribution through 50 mL aliquots during complete microwave extraction (until achieving burning point at total dryness of the matrix).

- ❖ No addition of water was required to perform the experiments (internal water works as the solvent of extraction).
- ❖ For **apple pomace**, and as observed with broccoli florets, a shrink cylinder shape (completly dry matter) at the end of the extraction was obtained.
- ❖ A constant 6 min time was required to obtain 50 mL aliquots after the initial and final heating periods.
- ❖ Good recoveries of material were observed during the initial five extraction cycles. The remaining period of time was necessary to achieve total dryness.

- ❖ **Spent coffee grounds** behave as a re-hydrate matrix, (water is added to the ground coffee when preparing espresso coffee) with very low recoveries associated.
- ❖ No visual changes are observed in the matrix (no shrinking behaviour).
- ❖ The flow behavior shows a decreasing tendency along time.
- ❖ With water:ethanol (50:50) co-addition a strong brown colour extract is recovered. Caffeine presence was detected at 280 nm.

- ❖ As a re-hydrated matrix like coffee, **brown algae**, shows flow behavior similar to spent coffee grounds, and also low recoveries associated.
- ❖ No visual changes are observed in the matrix (no shrinking behaviour).
- ❖ With water:ethanol (50:50) co-addition a strong green colour extract was recovered.

5.Conclusions

- ✓ **NEOS-GR**, a microwave hydrodiffusion and gravity oven, has been presented as a potential equipment to completely **dry matrices** with high humidity (therefore highly perishable), while under the scope of **green extraction technologies**, also recovering **hydrophilic (non-volatile) compounds** from these wet matrices using its own water.
- ✓ Good recoveries were observed when using high water content matrices, such as **apple pomace** and **broccoli**. However, when using re-hydrated matrices, such as **brown algae, *Pterospartum tridentatum* inflorescences**, and **spent coffee grounds** it was observed that the amount of material extracted is very low.
- ✓ The initial low recoveries, when using **re-hydrated matrices**, can be overcome by the co-addition of ethanol, allowing to obtain fractions rich in phenolic compounds, as well as brown compounds and caffeine in the case of **spent coffee grounds**.

References

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2. A. Filly, X. Fernandez, M. Minuti, F. Visinoni, G. Cravotto, F. Chemat, *Food Chemistry* (2014), 150, 193-198.
3. R. Vadivambal, D.S. Jayas, Biosystems Engineering (2007), 98, 1-16.

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