!SSE 2022

Scale-up study of a subcritical water extraction (SWE) laboratory system for *Gelidium sesquipedale* red algae residue valorization

E. Trigueros^a, P. Alonso-Riaño^a, C. Ramos^a, C. I. K. Diop^a, M.T. Sanz^a, S. Beltrán^a, <u>A.E. Illera^{a,*}</u>

University of Burgos, Biotechnology and Food Science Dept. (Chemical Engineering Section) Pza. Misael Bañuelos s/n 09001 Burgos (Spain).

*Corresponding author: aeillera@ubu.es

1. Introduction

Gelidium sesquipedale is a red alga used as the major seaweed resource in the Spanish agar industry, providing high-quality agar. After industrial agar extraction process, a solid residue that is usually disposed of, is generated. It contains high amounts of proteins with all essential amino acids and carbohydrates such as glucans, galactans or arabinans¹, what makes of it a valuable product. Therefore, its reincorporation in the industry as a value-added product brings an interesting solution. Traditional methods used for bioactive compounds extraction from different raw materials present numerous drawbacks, namely, time-consuming, costly to dispose of used products and harmful to the environment and human health². In this work, a different approach is proposed, and among green technologies, subcritical water extraction (SWE) stands out as a great alternative to traditional extraction processes. SWE consists of using hot pressurized water above its boiling point, 100 °C, and below its critical point, 374 °C. Water is kept in the liquid state due to the effect of high pressure, which causes many of the properties of water to change, such as density or dielectric constant^{3,4}. Water dielectric constant, decreases with increasing temperature, being similar to organic solvents, at 200-250 °C, and being able to selectively extract polar or non-polar compounds⁵. Previous studies have shown the capacity of this technology to recover the protein and polysaccharide fractions from red algae residue. In order to assess the feasibility of the industrial-scale up of this process, a pilot-scale SWE system was tested and compared with a laboratory SWE system.

2. Materials and Methods

The raw material used in this study was industrial solid residue from *Gelidium sesquipedale* after industrial agar extraction. Prior to extraction, the residue was dried and milled, obtaining dried macroalga residue (DMR). Only the fraction below 500 μ m particle size was used for SWE, due to the requirements of the pilot plant. DMR was extracted in laboratory- and pilot-plant scale SWE systems, both of them in a discontinuous mode, with reactors of 500 ml and 25 L, respectively. SWE was carried out at 130 and 175 °C.

Different analytical methods were applied for the comparison of the extraction yield of the two systems; such as, polysaccharide fraction identification and quantification, total protein content and free amino acids determination, and total polyphenol content (TPC) measurement.

3. Results and discussion

Galactose was mainly recovered as oligomer fraction with maximum yields of 71.4 % (36 minutes) and 74.5 % (45 minutes) for pilot and lab-scale, respectively (Figure 1a), and it showed the highest extraction yields among other polysaccharides. Arabinose showed a 62% yield in the pilot plant SWE system, but lower values were determined for glucans, with maximum yields of 9.5 % in both systems. The production of sugar-degradation and sugar-dehydration compounds was measured. Acetic acid was the degradation product that was formed preferentially, while sugar dehydration products content such as furfural and 5-hydroxymethylfurfural (HMF) was very low (Figure 1b).

!SSE 2022

For the extraction of the protein fraction, final extraction yields were close to 40 % in both systems (Figure 2a), while free amino acids content was higher in the laboratory scale (Figure 2b). The greatest extraction yield was accounted for the smallest amino acids, such as glycine, alanine and aspartic acid.



Figure 1. a) Sugars yield extraction and b) 5-hydroxymethylfurfural content in SWE at the different time intervals from DMR<500 at lab-scale: 130 (\circ) and 175 °C (Δ), and pilot-scale SWE system: 130 (\bullet) and 175 °C (Δ).



Figure 2. a) Protein yield extraction and b) free amino acids per gram of protein in SWE extracts collected at the different time intervals from DMR<500 at lab-scale: 130 (\circ) and 175 °C (Δ), and pilot-scale SWE system: 130 (\bullet) and 175 °C (Δ).

Differences in total polyphenolic compounds (TPC) extraction were observed between systems. Increasing TPC content with time was determined for lab-scale system, reaching 17.9 g/kg MDR, while in pilot system a plateau phase was observed after 36 minutes of extraction at 9.0 g/kg MDR. This difference could be attributed to differences in the operating temperature caused by the heating system in the pilot plant.

4. Conclusions

SWE has been proven to be an efficient technology for bioactive compounds recovery such as carbohydrates, protein and amino acids from algae residue. Scaling up of subcritical water system from laboratory to pilot scale resulted in good and reproducible results. Therefore, feasibility of industrial-scale subcritical water system through scaling-up from lab to pilot system has been showed, although further research is needed to improve the pilot plant design and assure equal working conditions in both systems.

References

- 1. E. Trigueros, M.T. Sanz, P. Alonso-Riaño, S. Beltrán, C. Ramos, R. Melgosa, J. Appl. Phycol. 2021, 33:1181-1194.
- 2. V. Camel, Analyst. 2021, 126, 1182-1193.
- 3. M. Herrero, A. Cifuentes, E. Iban, Food Chemistry 2006, 98, 136–148.
- 4. S.M. Zakaria, S.M. Mustapa Kamal, Food Engineering Reviews, 2016, 8, 23-34.
- 5. M. Plaza, M. Amigo-Benavent, M.D. Castillo, E. Ibañez, M. Herrero, Food Res. Int. 2010, 43: 2341-2348.