

## Supercritical CO<sub>2</sub> extraction of antioxidants from the shell of the Amazonian fruit *Garcinia macrophylla*

Bruno Nogueira<sup>2\*</sup>, Beatriz Díaz-Reinoso<sup>1</sup>, M<sup>a</sup> Dolores Torres<sup>1</sup>, Noelia Flórez-Fernández<sup>1</sup>, Alberdan S. Santos<sup>3</sup>, Renan C. Chisté<sup>2,4</sup>, Herminia Domínguez<sup>1</sup>

(1) CINBIO, Department of Chemical Engineering, Campus Ourense, Edificio Politécnico, Universidad de Vigo,

As Lagoas, 32004 Ourense, Spain

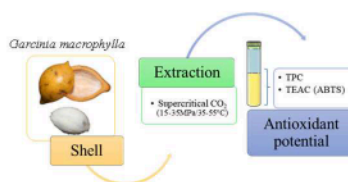
(2) Graduate Program of Food Science and Technology (PPGCTA), Institute of Technology (ITEC), Federal University of Pará (UFPA), 66075-110, Belém, Pará, Brazil.

(3) Laboratory of Systematic Research in Biotechnology and Molecular Biodiversity, Federal University of Pará (UFPA), 66075-110, Belém, Pará, Brazil.

(4) Faculty of Pharmacy, Federal University of Minas Gerais (UFMG), 31270-901, Belo Horizonte, MG, Brazil.

\*corresponding author: [bruno.nogueira@tucuruui.ufpa.br](mailto:bruno.nogueira@tucuruui.ufpa.br)

### GRAPHICAL ABSTRACT



### ABSTRACT

Amazonian biodiversity offers a huge potential for the study of bioactive compounds from fruits or vegetables. They can be a unique and renewable source of bioactive compounds such as phenolic compounds, carotenoids, vitamins, dietary fibers, among others. An example is *Garcinia macrophylla*, a perennial plant belonging to the genus *Garcinia*, the most representative of the Clusiaceae family, native to the Amazon region and adapted to acidic soils and humid climate [1]. The most studied parts are the stem bark and leaves, due to their antioxidant, antimicrobial, and antidiabetic properties [2]. The fruits are ovoid to ellipsoidal, with a yellow latex-containing rind, white tangy-sweet pulp, and 2 to 8 seeds. The chemical composition and biological properties of the fruits still require further research [3]. The chemical composition and biological properties of the fruits still requires further research [1]. The fruit shells are usually discarded but could be explored as a source of bioactives, biomaterials and bioenergy in the framework of a biorefinery. The initial extraction of bioactives would yield a valuable fraction with potential applications in food, nutraceutical and cosmetics sectors. In order to extract these compounds, ecofriendly extraction technologies could be applied, such as ultrasound assisted extraction, microwave assisted extractions, pressurized hot water extraction or supercritical carbon dioxide. The aim of this study was to explore the potential of supercritical carbon dioxide extraction for the extraction of phenolic compounds with antioxidant properties. *Garcinia macrophylla* Mars fruit shells, collected by extractivist communities in the Amazon, specifically in the state of Pará (Brazil), in February 2024, were dried, ground and stored in a dry dark place.

Conventional solvent extraction of the dried shells was performed with different solvents at a

liquid:solid ratio 20:1 (v:w) at 40 °C during 24 h on shaker protected from light to limit the possibility of oxidation. Supercritical fluid extraction of the dried shells (20 g), packed with glass beads, was performed in a 1 L cylinder extractor (Thar Designs SFE-1000F-2-C10, Pittsburgh, USA), equipped with two 0.5 L separators. The CO<sub>2</sub> was precooled with a circulating bath (PolyScience, USA, model 9506) before being pumped by a P-200A piston pump (Thar Design Inc., Pittsburgh, PA, USA) at 25 g CO<sub>2</sub>/min. Absolute ethanol was used as polar modifier and pumped by a HPLC pump (Scientific Systems, Inc., USA, model Series III) at a flow rate to achieve 10% (wt). The extract was collected in the first separator, operating at ambient temperature. Pressure and temperatures were studied in the ranges 15–35 MPa and 35–55 °C, respectively. The separator was further washed with absolute ethanol to recover the extract. Ethanol was removed in rotavapor and the resulting crude extracts were subsequently redissolved in absolute ethanol (10 mL) and stirring until complete dissolution. Samples were kept at 4 °C in darkness. Extraction yield values ranged from 5.6 - 8.5 g extract/100 g shells. The lowest extraction yield was obtained at 35 MPa and 35 °C. A positive influence of increasing pressure was noticed at 45 °C and 55 °C. At constant pressure, an increase in temperature has a positive effect in the global extraction yield except for the lower pressure, showing the crossover point phenomenon at around 20 MPa.

The data show that, under a pressure of 15 MPa, the highest phenolic compound content (204.23 mg GAE eq/g extract) was obtained at 35°C, decreasing to 183.45 mg/g at 45°C, and slightly increasing to 191.26 mg GAE eq/g extract at 55°C. At 25 MPa, the highest content (205.29 mg GAE eq/g extract) was observed at 45°C, with lower values at 35°C (171.02 mg GAE eq/g extract) and 55°C (166.67 mg GAE eq/g extract). Under 35 MPa, the values are high at 35°C (198.67 mg GAE eq/g extract) and 55°C (199.42 mg GAE eq/g extract), but lower at 45°C (174.99 mg GAE eq./g extract). As expected, increased pressure lowers the selectivity during the extraction of phenolics with supercritical CO<sub>2</sub> from *Garcinia macrophylla* shell. The extracts showed an ABTS radical scavenging capacity that ranged from 0.21 to 0.30 g Trolox eq/g extract, the highest value being obtained at 15 MPa and 45 °C. Additional studies are necessary to fully characterize the extracts for adding better understand and establish more precise extraction parameters.

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