**Optimizing Microwave-Assisted Synthesis of Carboxymethyl Tara Gum for Pharmaceutical Applications**

M. Pariguanaa,b, G. Cabrera-Barjasc, L. Gonzalezd, K. Varaprasadd Esteban F. Durán-Laraa\*.

a *Laboratory of Bio & Nano Materials, Drug Delivery and Controlled Release, Department of Microbiology, Faculty of Health Sciences, University of Talca, Talca 3460000, Chile.*

b *Doctorate in Science Mention R&D of Biactive products, Institute of Natural Resources Chemistry, University of Talca, Casilla 747, Talca 3460000, Chile.*

*C Facultad de Ciencias para el Cuidado de la Salud, Universidad San Sebastián, Campus Las Tres Pascualas, Concepción, Chile*

*d Nanotechnology and Advanced Materials Laboratory, Faculty of Engineering, Architecture and Design, University of San Sebastian, Concepción, Chile.*

*Email: eduran@utalca.cl*

**Summary**

Tara gum polysaccharides (TG) are natural biopolymers with significant potential for modification, making them particularly valuable for developing functionalized materials in the pharmaceutical industry [1]. Among these derivatives, carboxymethyl Tara gum (CMTG) stands out due to its high viscosity, which makes it an ideal candidate for controlled drug delivery applications [2]. This study focused on optimizing the heterogeneous synthesis of CMTG using microwave-assisted energy and comparing it with traditional synthesis methods [3].

The microwave-assisted synthesis of CMTG was adapted from a previously described dry synthesis approach [4]. TG (2g) was mixed with varying amounts of NaOH in a three-neck round-bottom flask containing 15 mL of 95% methanol (v/v) and processed in a UWave-2000 microwave oven (Sineo, China). The carboxymethylation reaction was conducted over 25 minutes at three different power levels: 450 W, 550 W, and 650 W. To determine the optimal ratio of TG to monochloroacetic acid (MCA) during microwave treatment, the reaction was tested under two different molar ratios: 1:10 and 1:12. The degree of substitution (DS) of the derivatives was determined using potentiometric titration and elemental analysis (CHONS), while FTIR, TGA, and SEM analyses were performed to characterize the CMTG samples.

A brief 25-minute microwave irradiation at 550 W efficiently produced a pale yellowish-white CMTG powder, significantly reducing reaction time compared to the traditional 5-hour method. The highest degree of substitution (DS) was achieved with a TG/MCA molar ratio of 1:12. The resulting CMTG exhibited excellent solubility in both distilled water and a 0.1 M aqueous NaOH solution. Notably, the microwave-assisted method yielded a higher DS (1.01) compared to the reflux method (DS 0.62).

The FTIR spectra of CMTG reveal characteristic vibrations of OH, CH2, and COO– groups, with bands at 3200-3400 cm−1, 2980 cm−1, and 1600 cm−1, respectively, indicating successful carboxymethylation. Additional peaks at 1030 cm−1 and 1415 cm−1 further confirm these modifications, with intensified absorptions related to COO– and CH2 compared to purified Tara gum. The TGA-DTA curves show that both purified Tara gum (GT) and CMTG undergo initial dehydration around 64.8°C, followed by more significant degradation between 235-350°C, resulting in a weight loss exceeding 60%. These findings suggest that CMTG derivatives exhibit greater thermal stability than GT, with a 20°C higher stability and reduced weight loss during the carbonization phase.

**Acknowledgement.** This work was supported by ANID FONDECYT REGULAR (Chile) through Project Nº 1210476 and ANID-FOVI (Chile) through Project Nº 230019 from Prof. Esteban F. Durán- Lara, G.C-B acknowledge Fondecyt Regular Nº 1221609.

**Reference**

[1] E. Moscoso-Moscoso *et al*., Polymers 16, (6) 1–19, 2024.

[2] M. B. Santos *et al*. Int. J. Biol. Macromol., 134, 595–603, 2019.

[3] D. M. Dos Santos *et al*. Carbohydr. Polym., 131, 125–133, 2015.

[4] Y. Gong, *et al*. Nanotechnology, 23, (29), 2012.