



**HAL**  
open science

## Valorization of silk by-products for lipopeptide surfactant production using green technologies

Lara Wehbe, Rémi Bascou, Erwann Guénin, Alla Nesterenko

### ► To cite this version:

Lara Wehbe, Rémi Bascou, Erwann Guénin, Alla Nesterenko. Valorization of silk by-products for lipopeptide surfactant production using green technologies. Green Extraction of Natural Products (GENP2024), Oct 2024, Compiègne, France. hal-04766036

**HAL Id: hal-04766036**

**<https://hal.science/hal-04766036v1>**

Submitted on 20 Nov 2024

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



## Valorization of silk by-products for lipopeptide surfactant production using green technologies.

Lara Wehbe <sup>(1)</sup>, Rémi Bascou <sup>(1)</sup>, Erwann Guénin <sup>(1)</sup>, Alla Nesterenko <sup>(1)</sup>

<sup>(1)</sup> Université de technologie de Compiègne, ESCOM, TIMR (Integrated Transformations of Renewable Matter), Centre de recherche Royallieu – CS 60 319 – 60 203 Compiègne Cedex.

Corresponding author: Lara Wehbe, [lara.wehbe@utc.fr](mailto:lara.wehbe@utc.fr)

**Keywords:** Silk biomass, protein extraction, lipopeptide, surfactant, mechanochemistry

### Objectives

Silk is defined as non-food biomass produced by cultured silkworms. Global silk cocoon production is approximately 800,000 tons per year<sup>1</sup>. Silks are composed of two types of proteins, fibrous protein called fibroin and a glue-like protein called sericin<sup>2</sup>. Traditionally, the main application of silk is the textile industry due to the luster and mechanical properties of fibroin fibers. The main objective of this work is the valorization of silk proteins as a non-food biomass for the development of bio-based lipopeptide surfactants. Generally, biobased surfactants are derived from agricultural biomass such as sugars, starches, vegetable proteins, and oils. The use of silk protein by-products allows to preserve agricultural biomass for food applications and to propose this new family of lipopeptides for non-food applications, especially cosmetics and detergents.

### Methods

The first step of silk lipopeptide production involves the separation of two silk protein fractions: fibroin and sericin by alkaline method and high temperature high pressure method (HTHP) respectively<sup>3, 4</sup>. Then, fibroin and sericin peptides were obtained by enzymatic hydrolysis. Finally, the grafting of the hydrophobic chain onto the silk peptides was carried out using novel alternative approach of acylation by mechanochemistry as green technology in solventless conditions. Two acylation methods were investigated: pre-activation of fatty acid by 1,1-carbonyldiimidazole (CDI) and amidation of a fatty ester. The optimization of the mechanochemical syntheses was carried out using model amino acids (leucine, glycine and serine) mainly present in the structure of silk. The degree of hydrolysis and the acylation rate (ratio of grafted amine functions) were estimated by spectrophotometry. The adsorption properties of the peptides and lipopeptide mixtures with different structure was then studied by surface tension measurements using the pendant drop method. Lastly, the green aspect of these syntheses was confirmed by green metrics calculation such as E-factor and the atom economy<sup>5</sup>.

### Results

The obtained results show that the two silk protein fractions can be efficiently separated. Silk sericin and fibroin fractions were hydrolysed using Alcalase® enzyme with a degree of hydrolysis varying between 20 and 60%. Moreover, the results of functionalization of silk peptides by mechanochemistry show very promising results with acylation rates varying from 50 to 70%. Silk lipopeptides with different fatty chains demonstrated a good

surface activity. Fibroin peptides and lipopeptides show better surface activity than those of sericin because of the hydrophobic nature of fibroin protein. The results confirm that the structure of the hydrophilic and hydrophobic parts of lipopeptides have a key role in their surface adsorption properties. Green chemistry parameters confirmed the “eco-compatibility” of new mechanochemical synthesis approach.

To conclude, the acylation of silk peptides through pre-activation by CDI and amidation strategies under solventless conditions appears promising for the development of lipopeptide surfactants. Mechanochemistry shows its efficiency in producing molecules of interest with good surface adsorption properties. Silk-derived lipopeptides, produced by mechanochemistry, have a significant potential to replace petroleum-derived amphiphilic molecules, for cosmetic, detergent and pharmaceutical applications.

- 1 A. Popescu, *Scientific Papers Series Management*, 2018, **18**, 385-400.
- 2 C. Vepari and D. L. Kaplan, *Prog. Polym. Sci.*, 2007, **32**, 991-1007.
- 3 D. N. Rockwood, R. C. Preda, T. Yücel, X. Wang, M. L. Lovett and D. L. Kaplan, *Nature Protocols*, 2011, **6**, 1612-1631.
- 4 R. Bascou, J. Hardouin, M. A. Ben Mlouka, E. Guénin and A. Nesterenko, *Materials Today Communications*, 2022, **33**, 104491.
- 5 S. Fadlallah, P. Sinha Roy, G. Garnier, K. Saito and F. Allais, *Green Chemistry*, 2021, **23**, 1495-1535.