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Lipase-catalyzed acylation of bio-oil enriched with levoglucosan: antibacterial and biosurfactant studies

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► **To cite this version:**

Marcelo Do Nascimento, Renato Froidevaux, Robert Wojcieszak, Ivaldo Itabaiana. Lipase-catalyzed acylation of bio-oil enriched with levoglucosan: antibacterial and biosurfactant studies. BiotechFrance, Jun 2022, Paris, France. hal-04562045

HAL Id: hal-04562045

<https://hal.univ-lille.fr/hal-04562045v1>

Submitted on 28 Apr 2024

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Introduction

Studies involving the transformation of lignocellulosic biomass into high value-added chemical products have been intensively conducted in recent years. Levoglucosan (1,6-anhydroglucopyranose (1)) is an anhydrous sugar that can be obtained by pyrolysis of cellulose.^[1] This anhydrous carbohydrate can be acylated to obtain carbohydrate fatty acid esters (CFAEs) (Figure 1), which can generate compounds with a hydrophobic-lipophilic balance (HLB) of great industrial interest as emulsifying agents, stabilizers in conventional systems and also promising biological activities.^[2-3] Here, these compounds were obtained via enzymatic acylation by biocatalysts such as lipases (triacylglycerol hidrolases, E.C. 3.1.1.3) of 1 that was commercially obtained (Start BioSci-ence®) and from cellulose pyrolysis, with different acyl donors (2) in batch and continuous flow. In this sense the objective of this work is to valorize lignocellulosic biomass through the synthesis of new levoglucosan esters, evaluating the regioselectivity of the enzymatic process in the presence of different acyl donors and investigating the biological activity of the final products.

Experimental

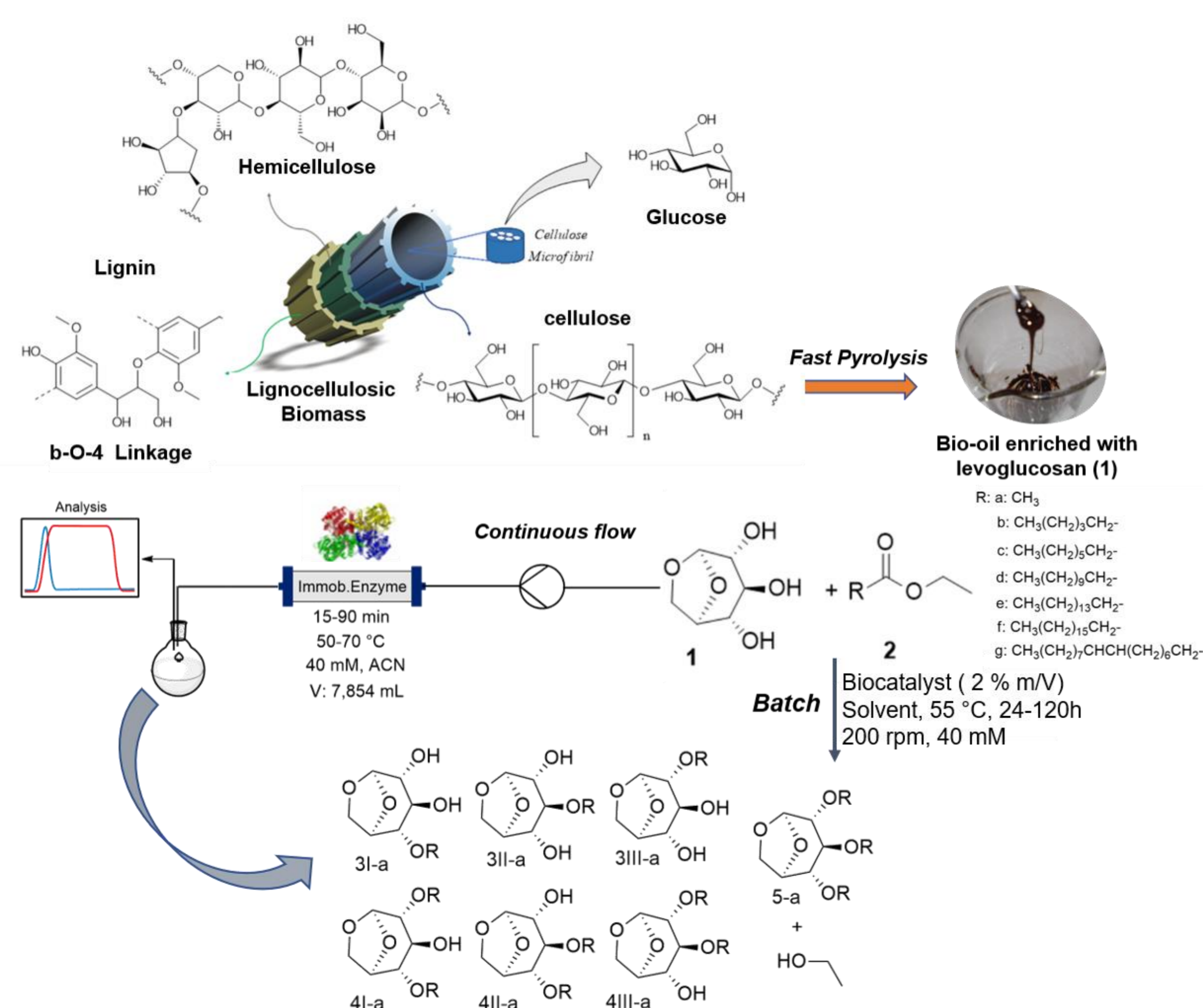


Figure 1: Schematic overview of the lignocellulosic biomass structure and transesterification reaction of levoglucosan with different acyl donors in batch and continuous flow.

Results and Discussion

In batch

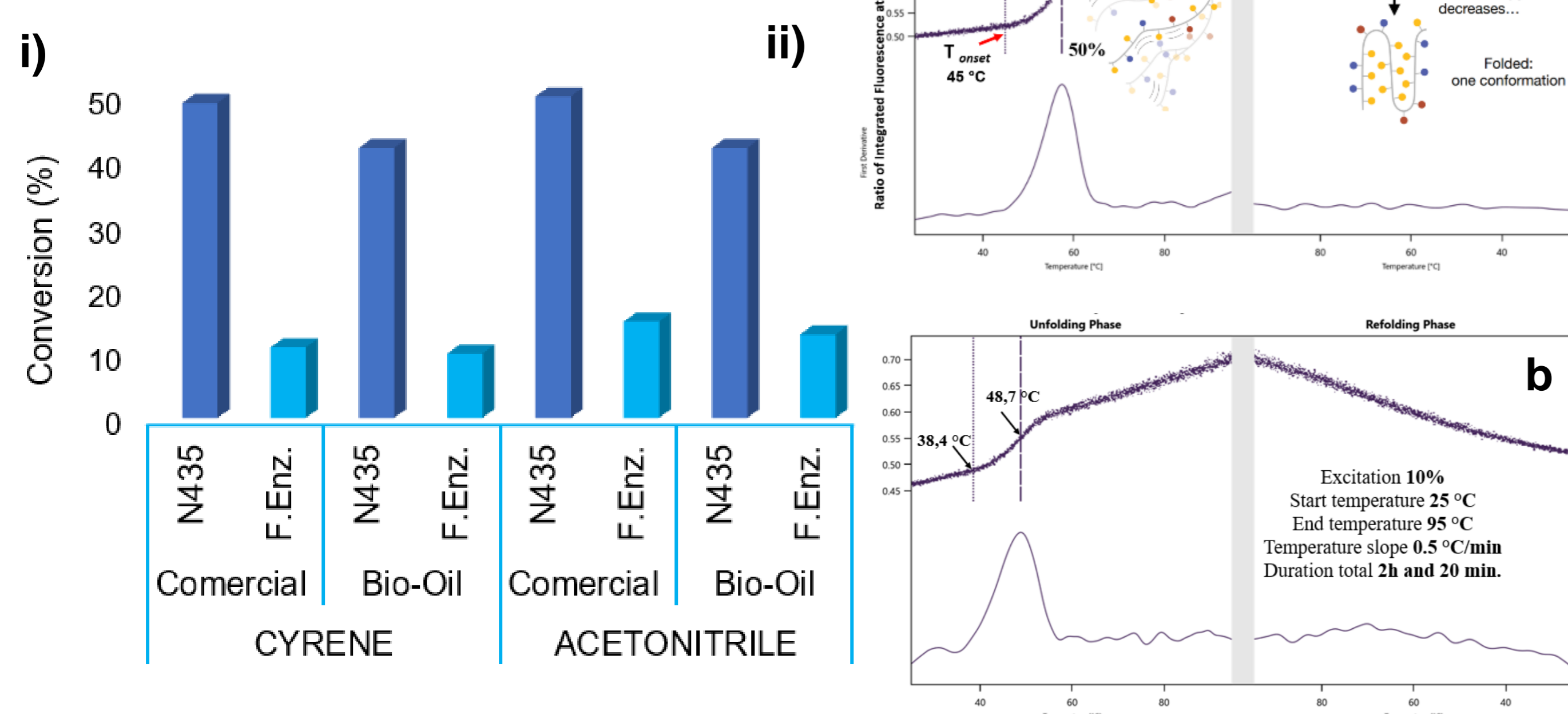


Figure 2: i) Comparing the influence on commercial levoglucosan source and obtained from bio-oil in the transesterification reaction between ethyl acetate and LG in different solvents: acetonitrile and cyrene, biocatalyzed by Novozyme 435 and lipase B from candida antarctica (free enzyme) and ii-a-b) Thermal analysis of free enzyme in a) acetonitrile and b) cyrene by nanoDSF (Prometheus NT.48).

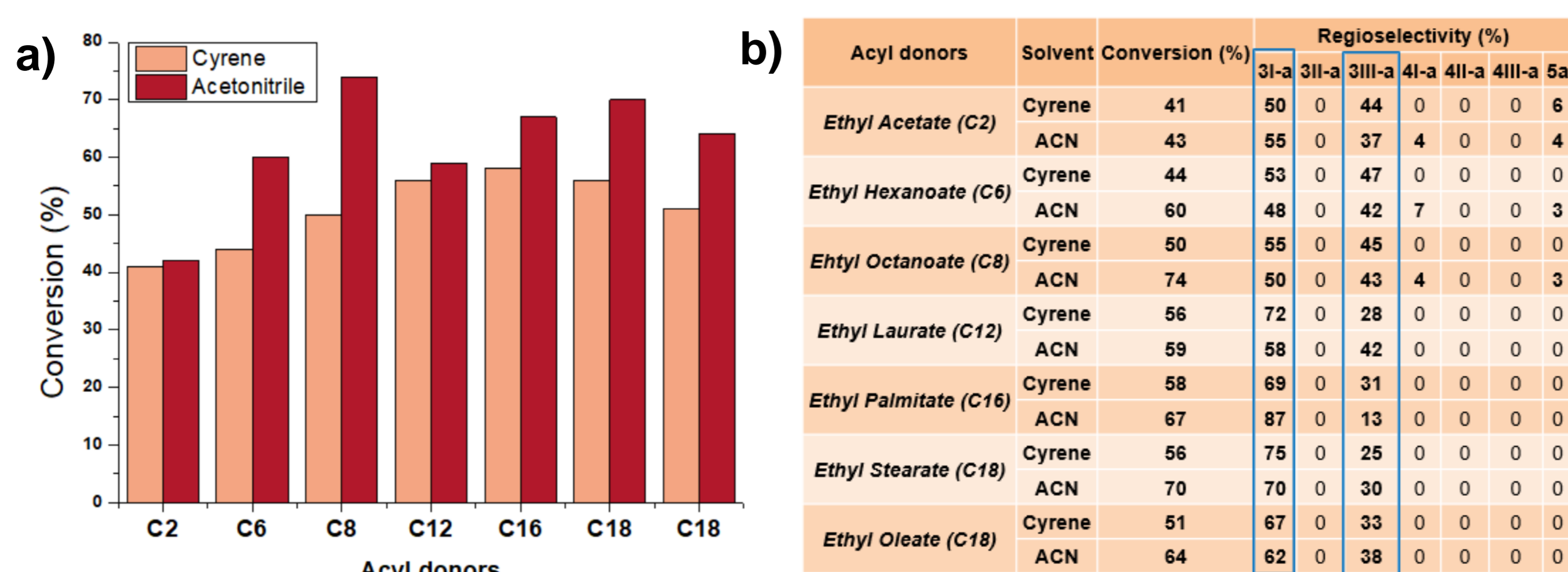


Figure 3: a) Comparing results between Cyrene and Acetonitrile for various ethyl acyl donors: Acetate (C2); Hexanoate (C6); Octanoate (C8); Laurate (C12); Palmitate (C16); Stearate (C18) and Oleate (C18) in the transesterification reaction of the levoglucosan in Bio-oil enriched and b) Regioselectivity.

In continuous flow

Through the experimental design, it was possible to optimize the reaction conditions, temperature and residence time obtaining a maximum conversion at 61 °C and 77 min (Figure 4).^[3]

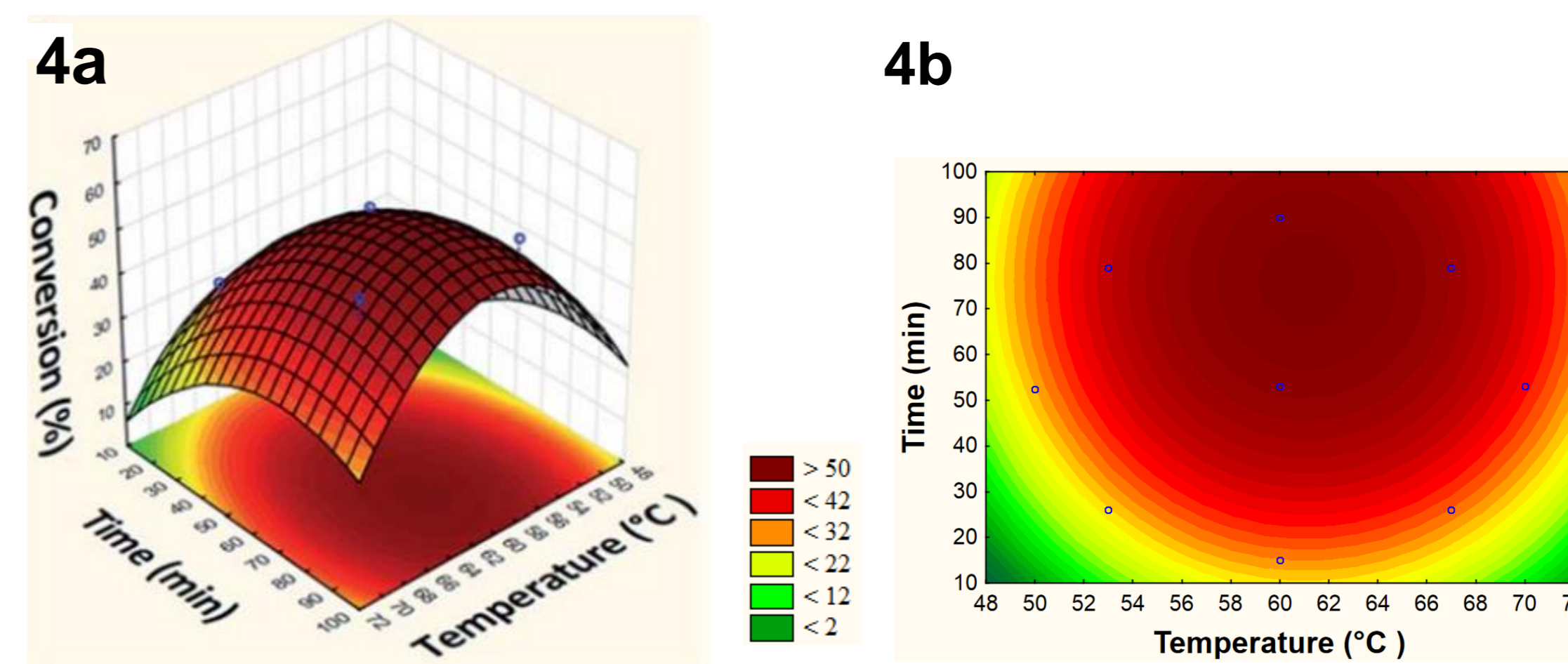


Figure 4: a) Response surface and b) level curve of the conversion in the model reaction of esterification of levoglucosan with lauric acid relating temperature and time.

Initial investigation of surfactant properties of obtained products has been carried out by measuring minimum interfacial tension (IFT^{min}). For a mixture of 4- and 2 O-lauryl-1,6-anhydroglucopyran (MONLAU), the minimum interfacial tension (IFT^{min}) obtained was 8.98 mNm⁻¹ and the critical micelle concentration (CMC) was 53 Mm (Figure 5).^[3]

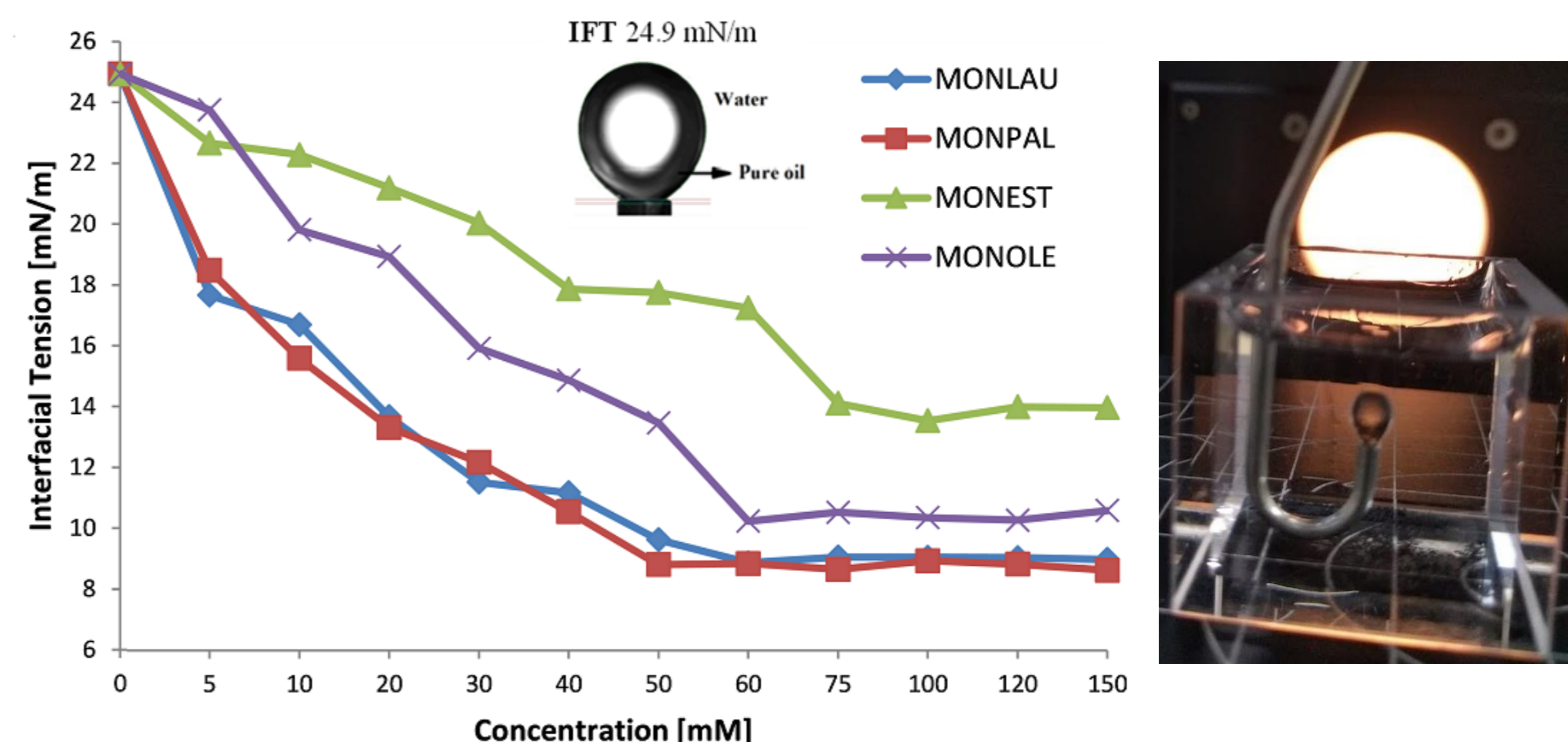


Figure 5: Relationship between the concentration of CFAs and interfacial tension.

Furthermore, promising data were obtained for minimal inhibitory concentration (MIC) and minimal bactericidal concentration (MBC) of MONLAU against *Staphylococcus aureus* strains at 0.25 Mm (Table 1).^[3]

Table 1: Minimal inhibitory concentration (MIC) and minimal bactericidal concentration (MBC) of products and substrates against *Staphylococcus aureus* strains

Compound	ATCC 25923 MSSA	ATCC 29213 MSSA	ATCC 33591 MRSA	517 MRSA
MONLAU	0.25 mM	0.25 mM	0.25 mM	0.25 mM
MONEST	> 1.0 mM	> 1.0 mM	> 1.0 mM	> 1.0 mM
MONPAL	> 1.0 mM	> 1.0 mM	1.0 mM	> 1.0 mM
MONOLE	1.0 mM	0.5 mM	0.25 mM	1.0 mM
Lauric Acid	0.5 mM	0.5 mM	0.5 mM	0.5 mM
Levoglucosan	> 1.0 mM	> 1.0 mM	> 1.0 mM	> 1.0 mM
Methicillin	0.5 µg.mL ⁻¹	0.25 µg.mL ⁻¹	4.0 µg.mL ⁻¹	4.0 µg.mL ⁻¹

a) minimal bactericidal concentration (MBC). MSSA: methicillin-sensitive *Staphylococcus aureus*. MRSA: methicillin-resistant *Staphylococcus aureus*.

Conclusion

In this work, it was possible to maximize the synthesis of CFAEs in continuous flow and also for the batch process through studies of different solvents and thermostability for free enzymes under different conditions. In addition, CFAEs were applied in surface activity tests through the analysis of minimum interfacial tension and in biological tests through the evaluation of antibacterial activity in species of *Staphylococcus aureus*, the clinical strain was isolated from patients of Clementino Fraga Filho University Hospital (UFRJ-RJ-Brazil) and three strains of American Type Culture Collection (ATCC).

References

¹ Itabaiana Junior, *et al.* Levoglucosan: A promising platform molecule? *Green Chem.*, 22 (18), 5859–5880. ² Galletti, P., *et al.* Enzymatic acylation of levoglucosan in acetonitrile and ionic liquids. *Green Chem.*, 9 (9), 987–991. ³ Avelar Do Nascimento, *et al.* Lipase-catalyzed acylation of levoglucosan in continuous flow: antibacterial and biosurfactant studies. *RSC Adv.*, 2022, 12, 3027–3035.