Supporting Information

Microwave barrel reactor for trimethylolpropane oleate synthesis by *Candida antarctica* lipase in a biphasic non-solvent process

Manuel Happe[1], Pascal Grand[2], Sébastien Farquet[2], Sandrine Aeby[3], Jean-Claude Héritier[2], François Corthay[2], Eric Mabillard[2], Roger Marti[3], Ennio Vanoli[3], Alain-François Grogg[1], Samuel Nussbaum[4], Alain Roduit[2], François Tièche[4], Sam Salem[5], Carole Constantin[1], Esther Schmitt[1], Silvan Zahno[2], Christoph Ellert[2], Ahmed Habib[1], Julien Wyss[2], Fabian Fischer[1]*

1) Institute of Life Technologies, University of Applied Sciences Western Switzerland, HES-SO Valais, Sion. Route du Rawyl 64, 1950 Sion, Tel. +41 (0) 27 606 86 58, email: Fabian.Fischer@hevs.ch.
2) Institute of Systems Engineering, University of Applied Sciences Western Switzerland, HES-SO Valais, Sion.
3) Institute of Chemistry University of Applied Sciences Western Switzerland, HES-SO Fribourg, Fribourg.
4) Information and Communication Systems Institute, University of Applied Sciences Western Switzerland, HE-arc, St Imier.
5) Institute of Economics, University of Applied Sciences Western Switzerland, heig-vd, Yverdon.

Supporting Information

Mixing modes of the microwave barrel reactor 2

Atrefacts in thermal imaging 2

Rapid ^{1}H-NMR method to determine reaction progress 3

Mass spectra of a lubricant mixture 3

Melting and fusion points of the biolubricants 4

Viscosity of the biolubricants 4

Project Video 5

Supporting Information

Mixing modes of the microwave barrel reactor

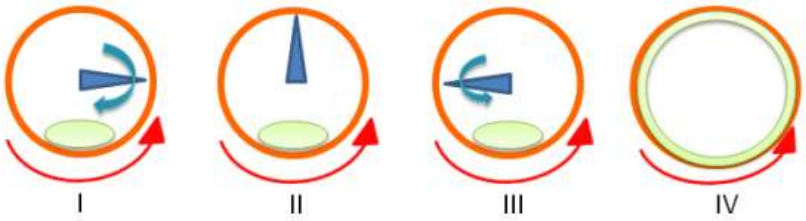

Figure S1: Tube and stirrer rotations (I-III) and the centrifugation mode (IV) for even large surface microwave irradiation.

Atrefacts in thermal imaging

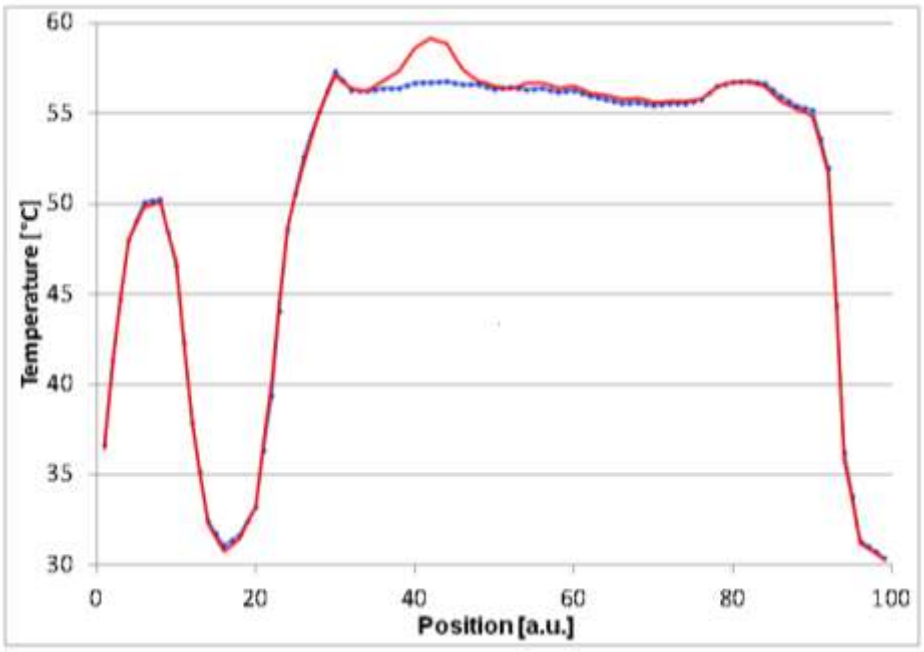

Figure S2: Temperature distribution obtained by a vertical scan of the horizontal reactor tube. An artificial temperature increase is observed between position 37 and 47 [a.u.] (red line). The infrared heat emission is reflected on the ceiling of the Faraday cage and causes the impression that the barrel surface is more heated in this zone even though the reactor tube is in constant rotation. The blue line is recorded by shielding this reflection, which eliminates the arte-fact.

Supporting Information

Rapid ^{1}H-NMR method to determine reaction progress

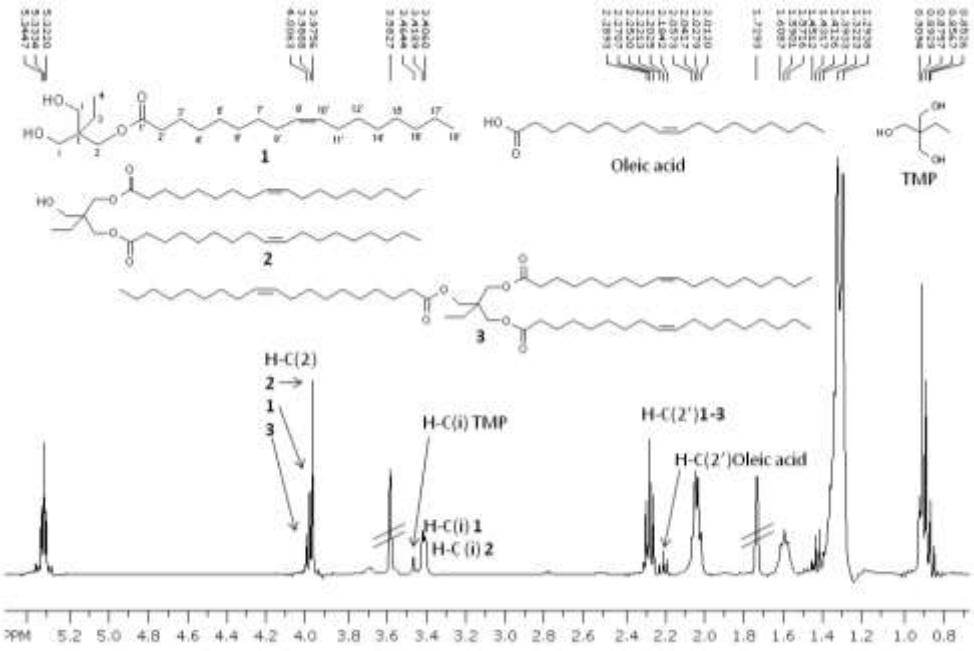

Figure S3: ^{1}H-NMR (400 MHz THF-$_{d8}$) of a reaction mixture containing three trimethyolpropane oleate lubricants **1-3**, unconverted TMP and oleic acid. THF-$_{d8}$ separates well all relevant chemical shifts. Oleic acid conversion was quantified by the H-C(2') triplet integral at 2.2 ppm as this triplet shifts upon esterification to 2.25 ppm providing also the overall yield. The TMP H-C(2) singlets at 3.45 ppm shifted upon esterification to 3.4 ppm as a broader singlet H-C(i) **1-2** and to ~4 ppm H-C(2). Here the product distribution was determined, the diester (**2**) is found at 3.98 ppm, the monoester (**1**) 3.99 ppm and the triester (**3**) at 4.01ppm.

Mass spectra of a lubricant mixture

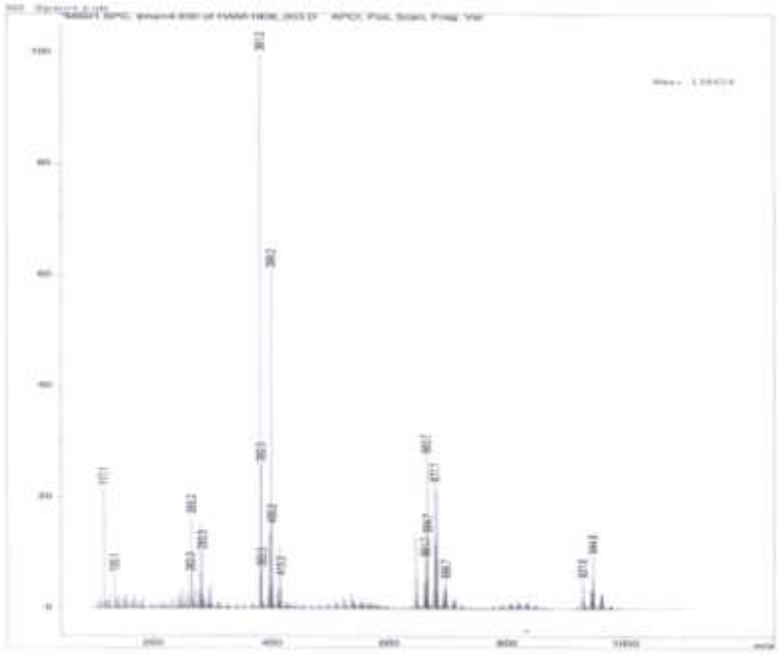

Figure S4: APCI-MS of trimethylolpropane oleates **1-3** mixture recorded from a 1:1 TMP/oleic acid mixture using 90% pure oleic acid that causes multiple molecular mass peaks.

Supporting Information

Melting and fusion points of the biolubricants

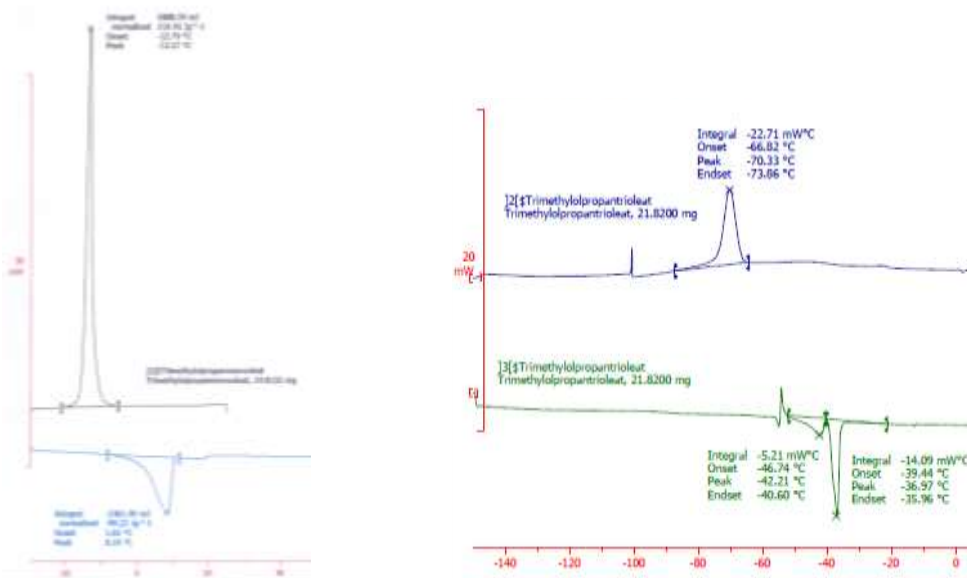

Figure S5: Crystallization and fusion points of trimethylolpropane monooletate **1** (left) and trimethylolpropane trioleate **3** (right) recorded by DSC.

Viscosity of the biolubricants

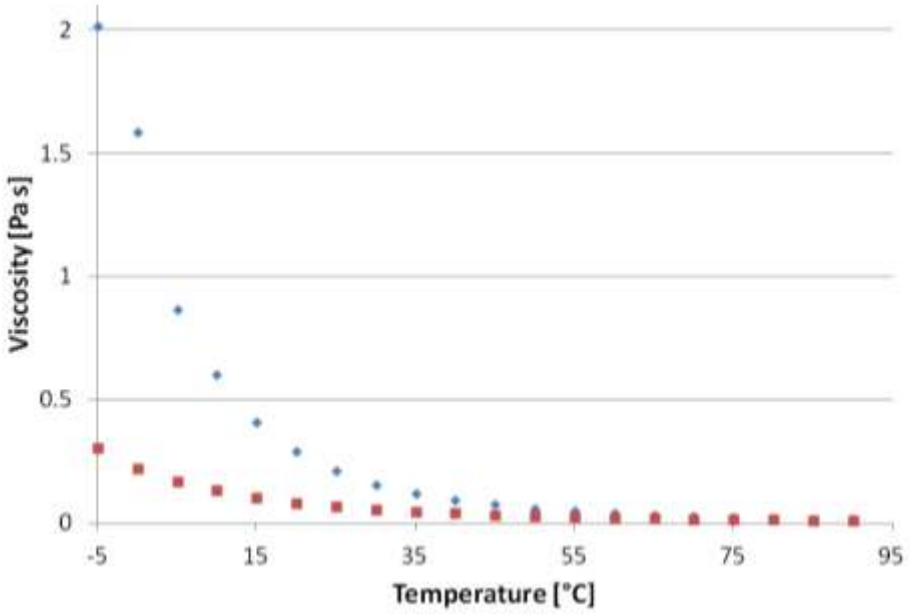

Figure S6: Shear viscosity of trimetylolpropane monooleat **1** and trimethylolpropane trioleate **3**. At lower temperature the viscosity is different and becomes equal above 75°C.

Supporting Information

Project Video

Figure S7: A video about the Microwave Barrel Reactor is available on the internet site of the University of Applied Sciences Western Switzerland (www.hesso.ch) or directly on Youtube; (www.youtube.com/watch?v=0MD2i7ErQKo or search for ECO^{2}BioPro ST").