Supporting Information

“Sweet Silicones”: Biocatalytic Reactions to Form Organosilicon Carbohydrate Macromers

Bishwabhusan Sahoo¹, Kurt F. Brandstadt²,
Thomas H. Lane², and Richard A. Gross^{1*}

*¹NSF IUCRC for Biocatalysis and Bioprocessing of Macromolecules,
Othmer Department of Chemical and Biological Sciences and Engineering,
Polytechnic University, Six Metrotech Center, Brooklyn,
New York 11201 [<http://chem.poly.edu/gross/>]*

²Dow Corning Corporation, Midland, Michigan 48686

Reaction of Ethyl glucoside with PDMS using novozym 435 as catalyst:

In a two-neck round-bottom flasks heated by an external oil bath (70°C), PDMS diacid and α,β -ethylglucoside were added with a 1:2 molar ratio and stirred with a magnetic stirring. After these components formed a monophasic mixture, dried N435 was added (N435 to the reactants was 1:10 w/w), the reaction mixture was maintained at 70°C for 2 hrs, and then maintained in-vacuo (65 mmHg) with magnetic stirring for an additional 34 hours. The reactions were monitored by thin layer chromatography (TLC) using silica gel plates (20x20cm, Silicagel 60 F254, EMD Chemicals Inc.) and chloroform/methanol mixtures (9:3 or 9/1 v/v) as mobile phase. After reactions, the mixtures were filtered to remove N435 and products were purified by flash silica gel column chromatography (silicagel 60, Aldrich). In a typical separation, 50g of silica gel was used to pack a glass column (5cm x 50cm) in the eluent (CHCl₃/MeOH mixture). Then, 200 mL of eluent was run through the column before the crude compound (500mg) dissolved in a minimal amount of eluent was loaded onto the top of the column silica bed. Different fractions were subsequently eluted (1mL/min) and monitored by thin layer chromatography (TLC). Fractions containing the purified compounds were pooled together, and the solvent was evaporated to give the pure product. The chromatographic separations of products from esterifications of 1,3-bis(3-carboxypropyl)tetra-methyldisiloxane ($x=0$) and the corresponding higher molar mass PDMS diacids ($x_{av}=7$ and 65) were performed using chloroform:methanol mixtures of 9:2 and 9.5/0.5 v/v, respectively.

Figure S-1. ^{13}C DEPT NMR (DMSO- d_6 , 75.5 MHz) spectra of 1,3-bis(1'-ethylglycosyl-6'-propionate) tetramethyldisiloxane (enlarged form).

Figure S-2 : TGA spectra of the 1,3-bis(carboxypropyl)-tetramethyl Disiloxane

Figure S-3: TGA spectra of the 1,3-bis(2'-ethylglycosyl-6'-propionate)tetramethyl disiloxane

Figure S-4: DSC spectra of the 1,3-bis(carboxypropyl)-tetramethyl Disiloxane

Figure S-5: DSC spectra of the 1,3-bis(2'-ethylglycosyl-6'-propionate)tetramethyl disiloxane