Synthesis of a H-Sulfo-POSS catalyst and application in the acetalization of glycerol with 2-butanone to yield a biofuel additive

*Viktor Söderholm[a], Jesús Esteban[b], Dieter Vogt[a]**

[a]*Laboratory of Industrial Chemistry, Department of Biochemical and Chemical Engineering, Technical University of Dortmund, Emil-Figge-Str. 66, D-44227 Dortmund, Germany*

[b] *Department of Chemical Engineering and Analytical Science, The University of Manchester, Manchester M13 9PL, United Kingdom*

[*]*Corresponding author: dieter.vogt@tu-dortmund.de*

Supplementary Information

Table of contents:

1. Quantitative evaluation of reaction by ^{1}H-NMR

The reaction mixture was analyzed by ^{1}H-NMR for quantitative determination of ketal product yield as reported previously 1.[1] A spectrum from a typical reaction mixture containing glycerol, butanone, ethanol and the product ketals is displayed in Figure SI1 with a proper identification of the peaks and the signals.

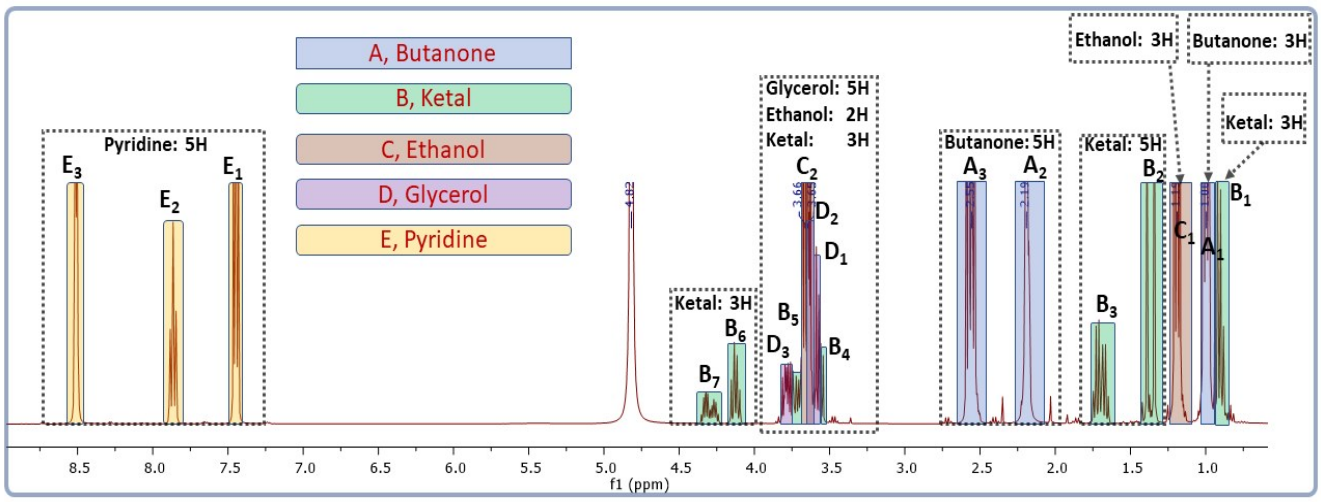

Figure SI1. ^{1}H-NMR spectrum of a typical reaction mixture from the ketalisation of glycerol by 2-butanone. The different peaks have been colored and labelled according the reaction mixture component that it is associated with.

By normalizing the integrals of the peaks related to the different compounds in the reaction mixture relative to product peak which has a known number of protons, the relative molar ratio between each compound and the product can be directly calculated. The signal labelled as "B_2" in the graphic was chosen as it is the product peak with highest number of associated protons. Yield can then be calculated based on the integral relationship between this "B_2" peak and peaks associated with either glycerol or butanone.

2. ^{1}H-NMR spectroscopic characterization of the catalysts

Figure SI2 depicts representative ^{1}H-NMR spectra of the synthesized catalysts.

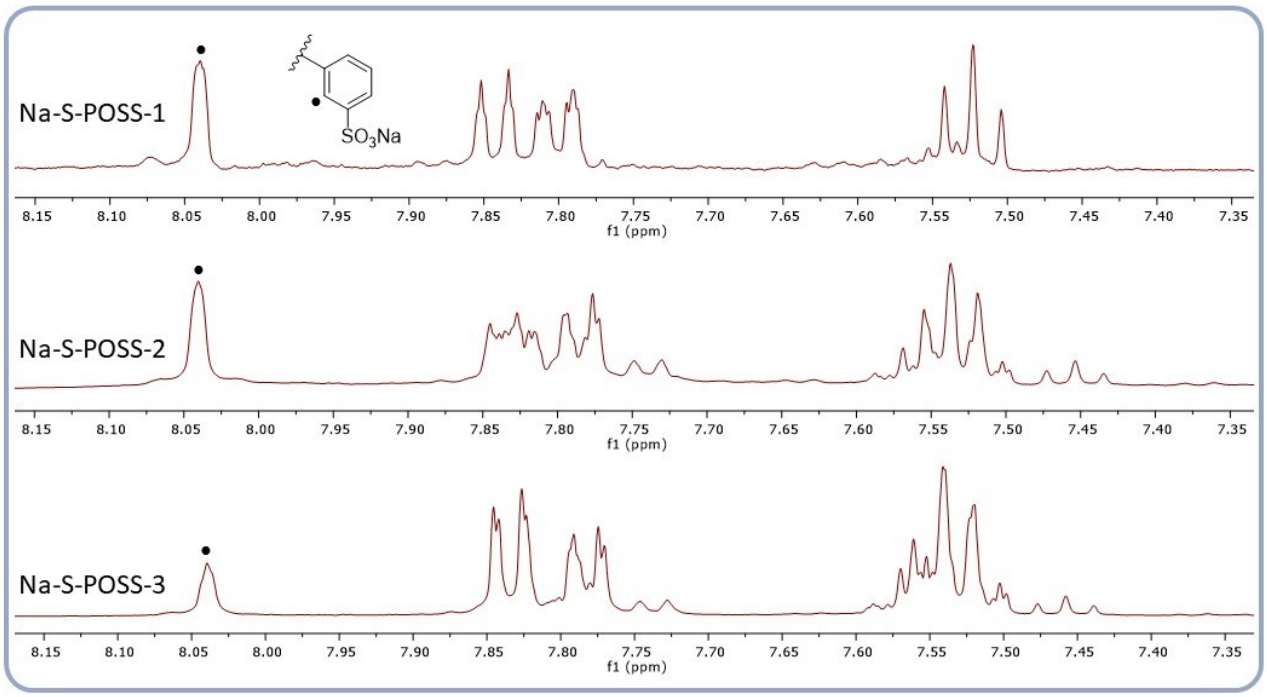

Figure SI2. ^{1}H-NMR of the sodium salts of S-POSS (Na-S-POSS-1, -2 and -3) dissolved in D_2O.

REFERENCE

1. J. Esteban, F. Garcia-Ochoa and M. Ladero, *Green Process. Syn.*, 2017, **6**, 79-89.