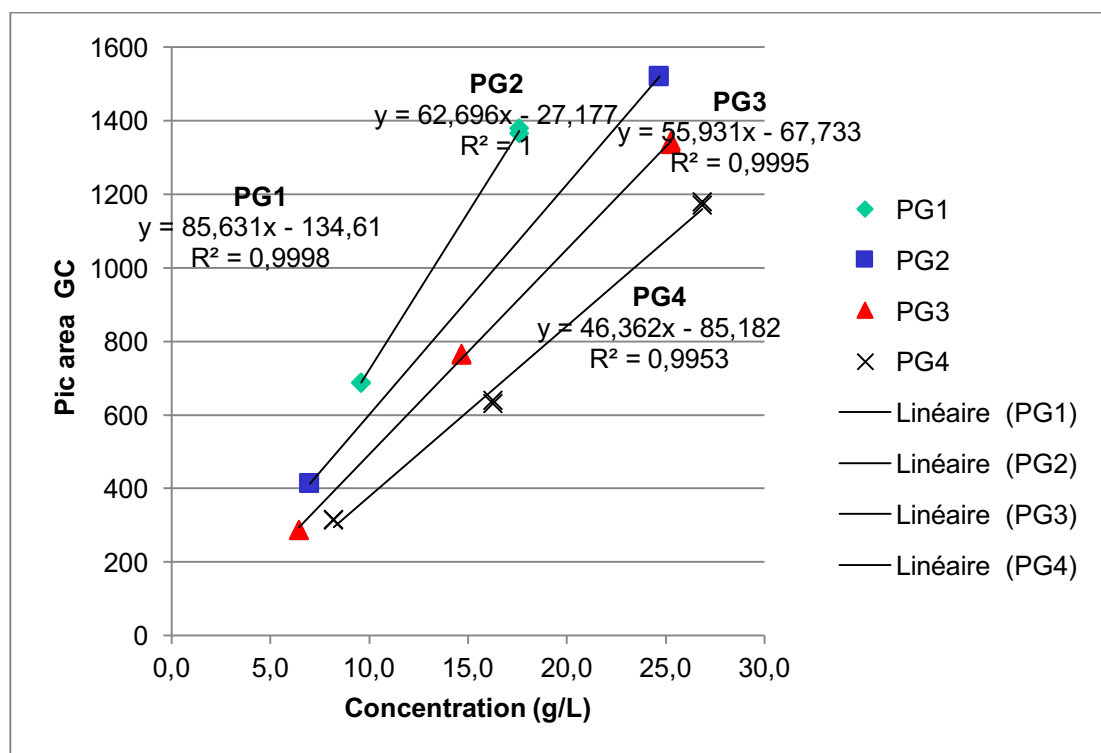


# Microwave-Assisted Continuous Flow for the Selective Oligomerization of Glycerol: Supporting Information

Rémi Nguyen, Nicolas Galy, Fatmah Ali Alasmary, and Christophe Len\*

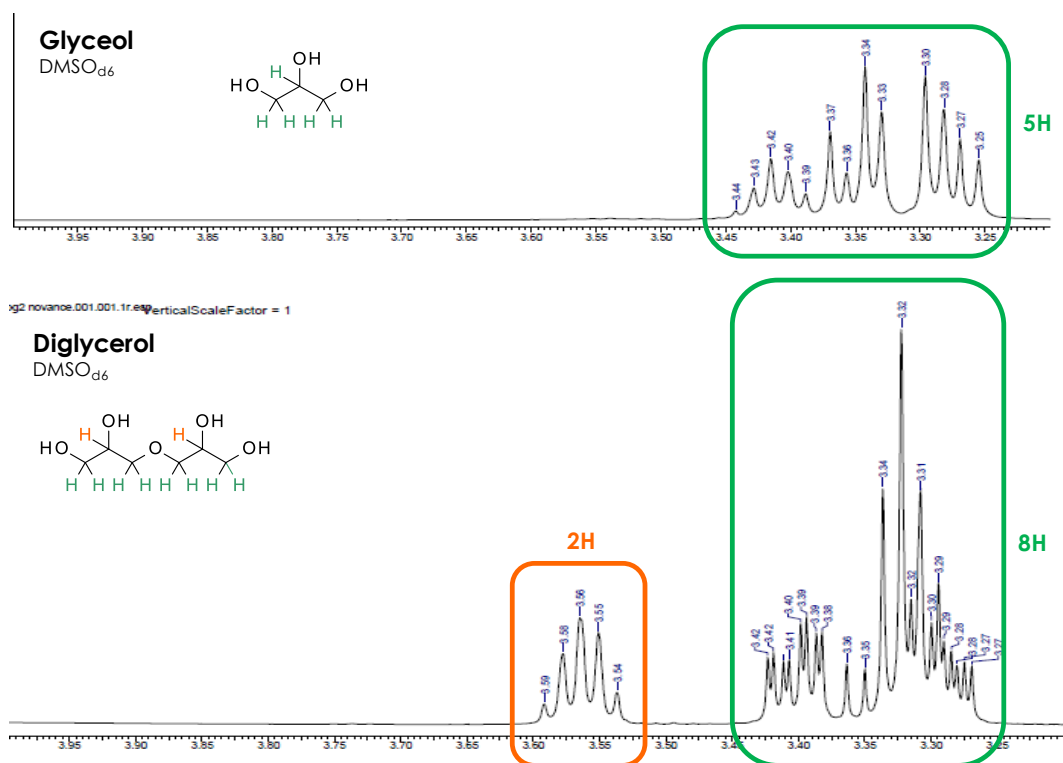


**Fig. S1.** Calibration curves for pure standard glycerol oligomers.

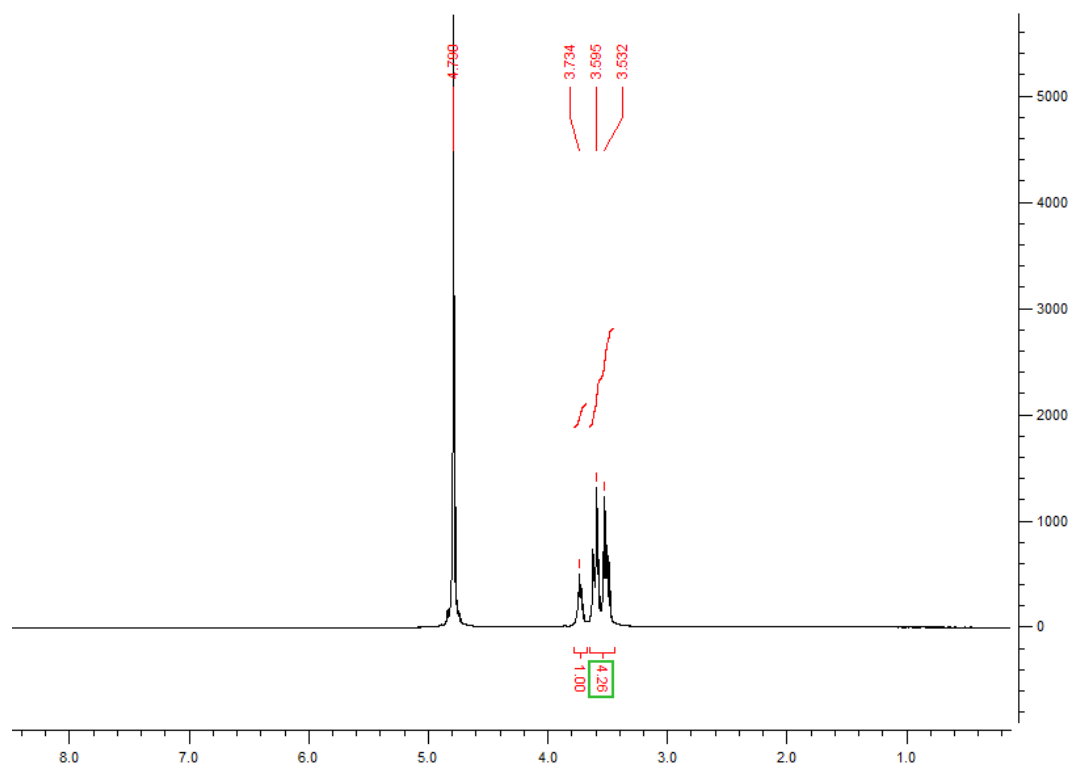
**Table S1.** Preliminary temperature study for continuous flow oligomerization of glycerol under microwaves.

Flow mode	Temperature	Conversion
1 mL/min	200 °C	0% (NMR)
1 mL/min	230 °C	1.1% (NMR)
1 mL/min 12 cycles (16h)	230 °C	8.0% (NMR)
1 mL/min	238 °C	6.5% (NMR)*

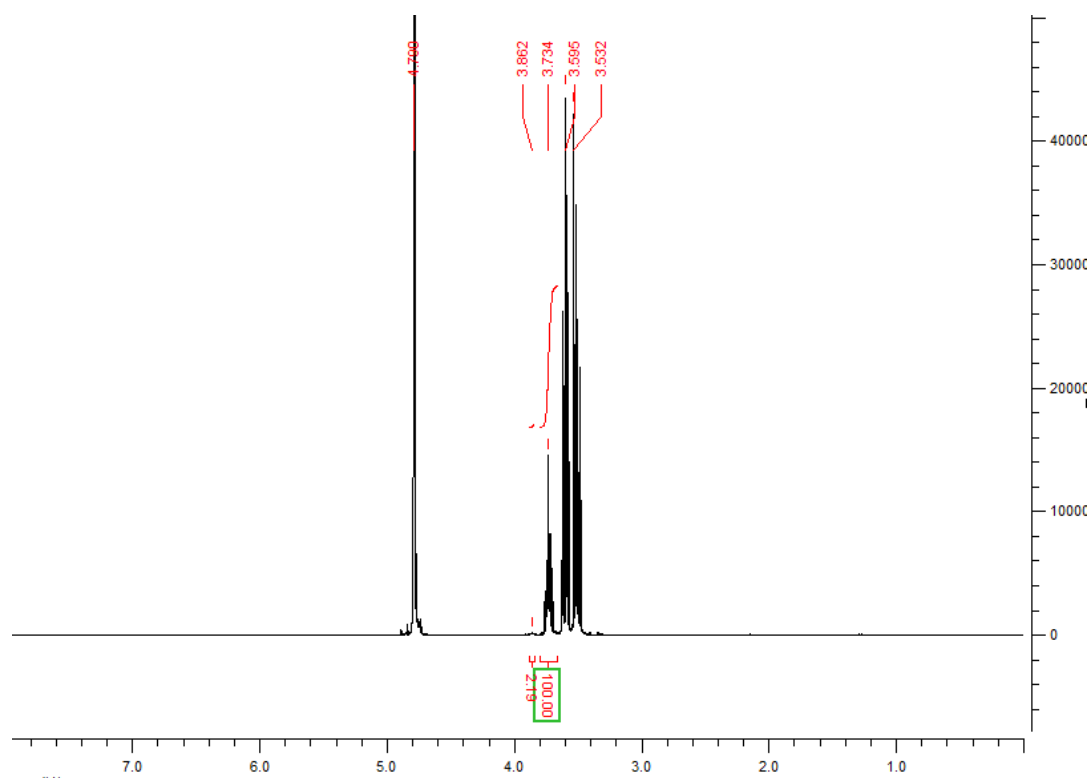
\*NMR is not accurate as other oligomers are formed



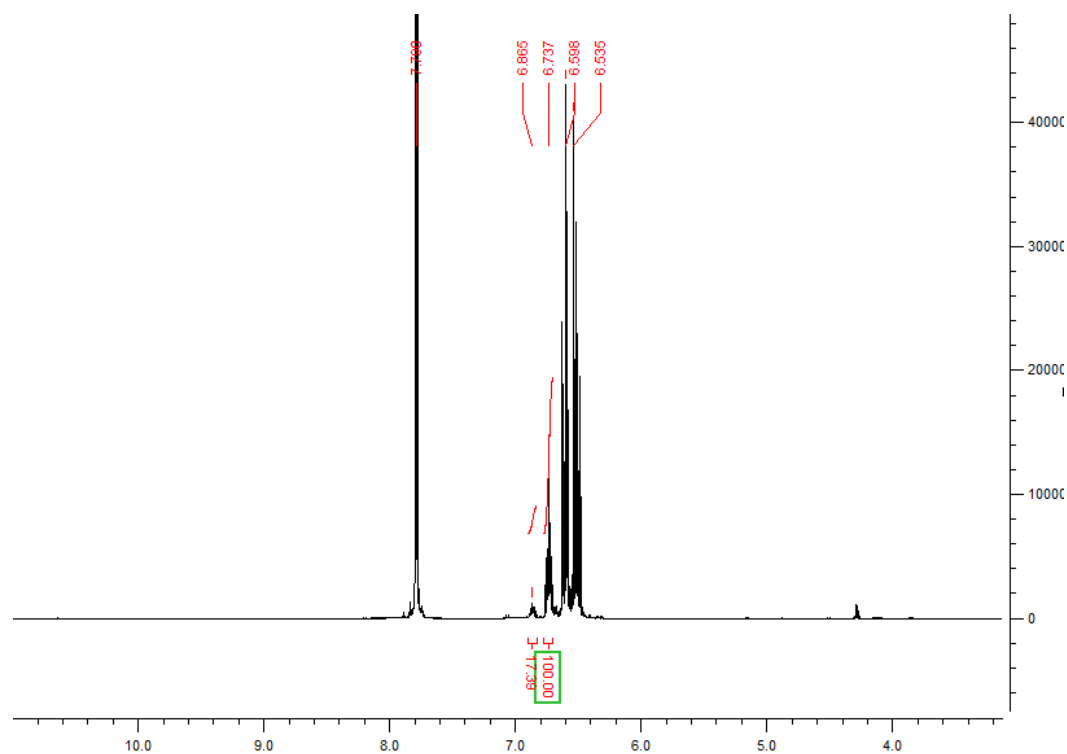
**Fig. S2.**  $^1\text{H}$  NMR (DMSO) signals of glycerol and diglycerol.



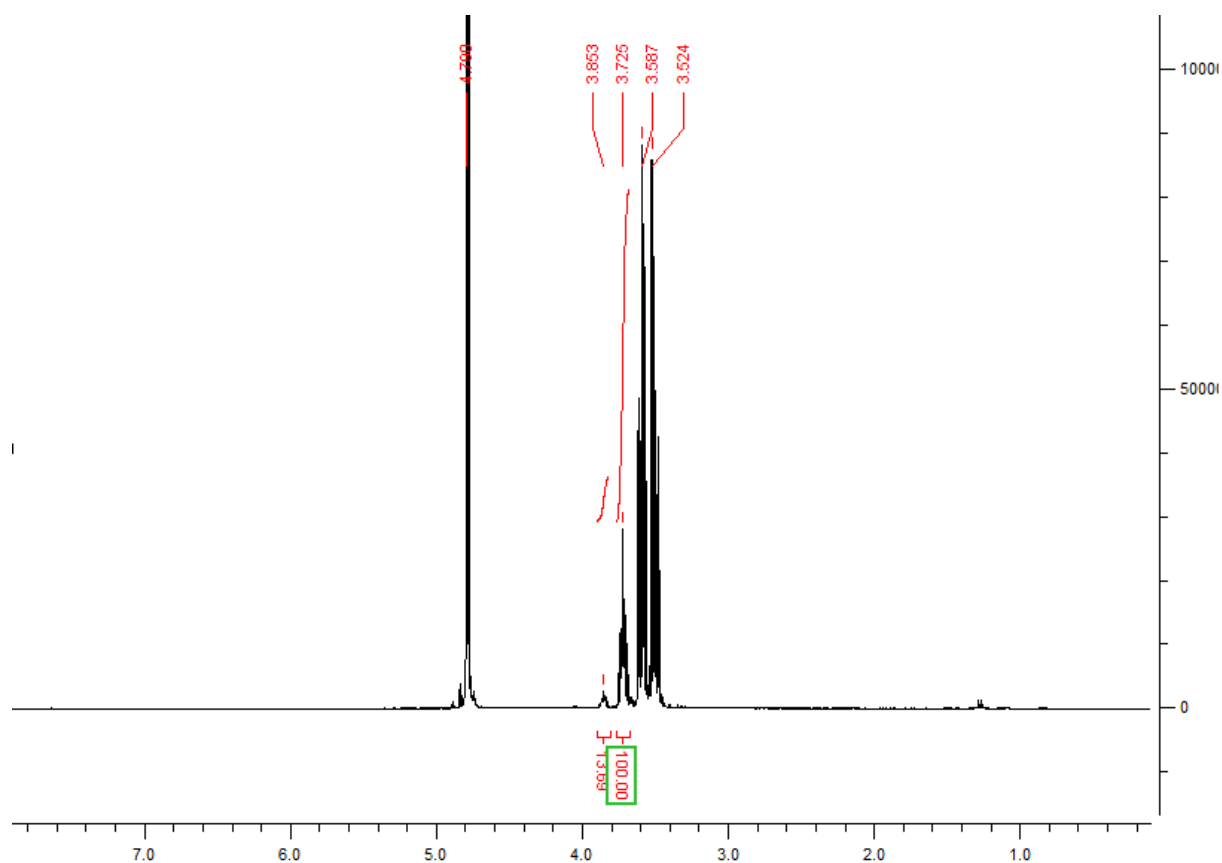
**Fig. S3.**  $^1\text{H}$  NMR (D<sub>2</sub>O) spectra of oligomerization of glycerol by microwaves at 200 °C with 1 mL/min continuous flow. 100 % glycerol is observed (4.73 ppm, 1H) with no PG2 signal.



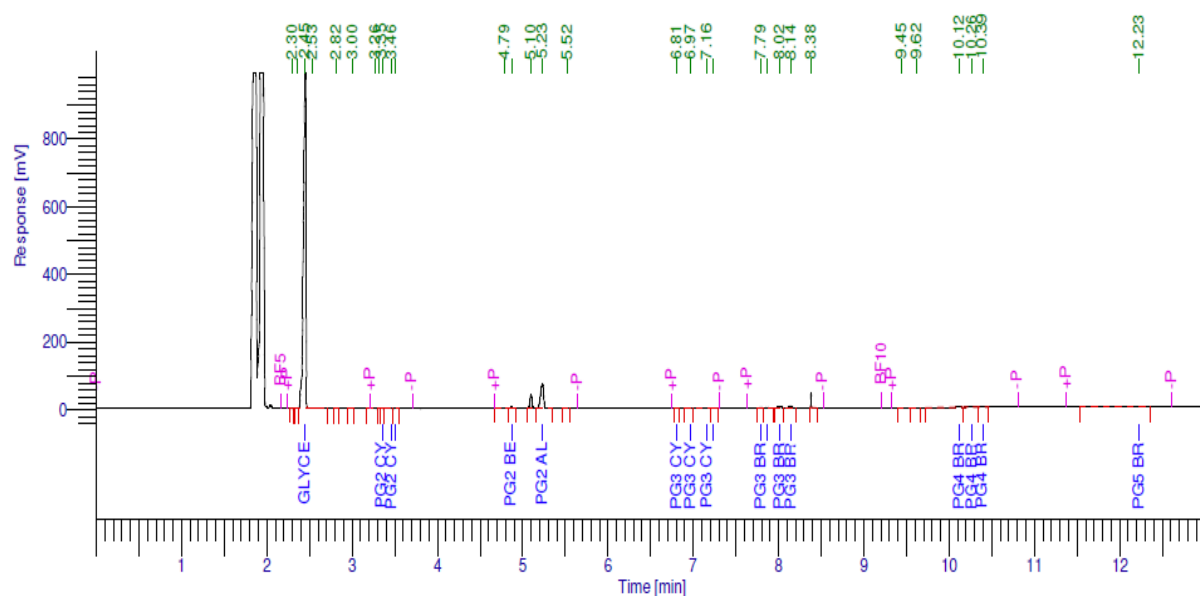
**Fig. S4.** <sup>1</sup>H NMR (D<sub>2</sub>O) spectra of oligomerization of glycerol by microwaves at 230 °C with 1 mL/min continuous flow. 1 % of diglycerol (4.86 ppm, 2H) is observed.



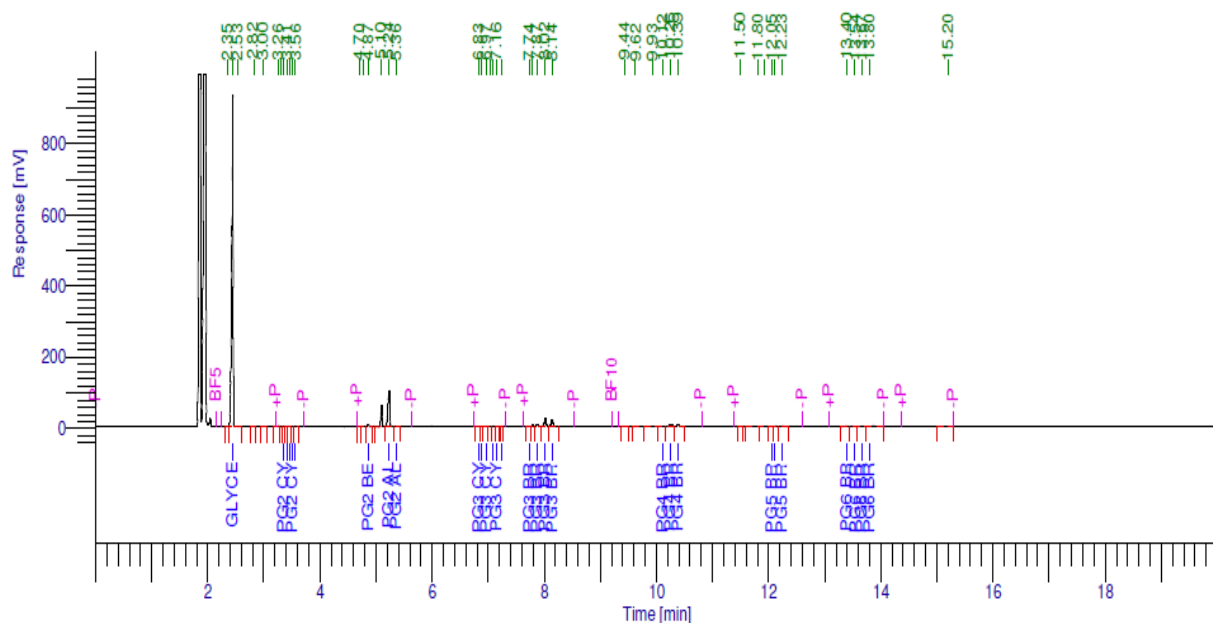
**Fig. S5.** <sup>1</sup>H NMR (D<sub>2</sub>O) spectra of oligomerization of glycerol by microwaves at 230 °C after 12 cycles at 1 mL/min continuous flow. 8 % of diglycerol (4.86 ppm, 2H) is observed.



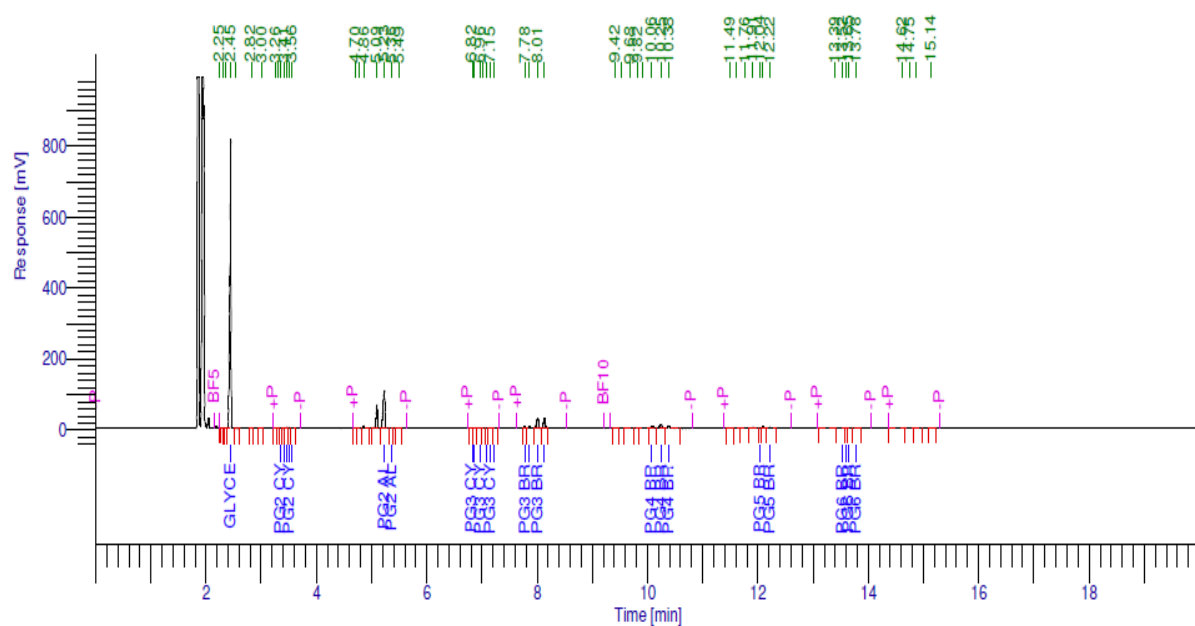
**Fig. S6.**  $^1\text{H}$  NMR ( $\text{D}_2\text{O}$ ) spectra of oligomerization of glycerol by microwaves at  $238^\circ\text{C}$  with 1 mL/min continuous flow. 6.5 % of diglycerol (4.86 ppm, 2H) is observed, but due to other glycerol oligomers formation, NMR is not accurate as quantitative tool.



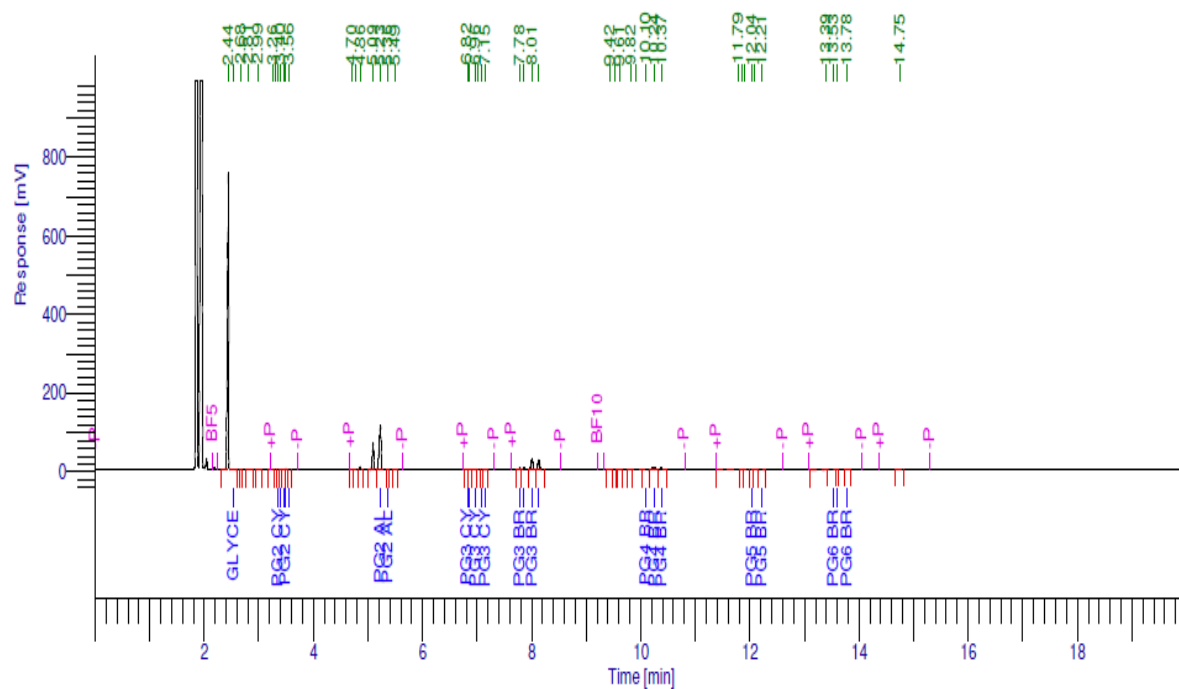
**Figure SI.7:** GC chromatogram of glycerol oligomerization by microwaves at  $238^\circ\text{C}$  with 1mL/min continuous flow.



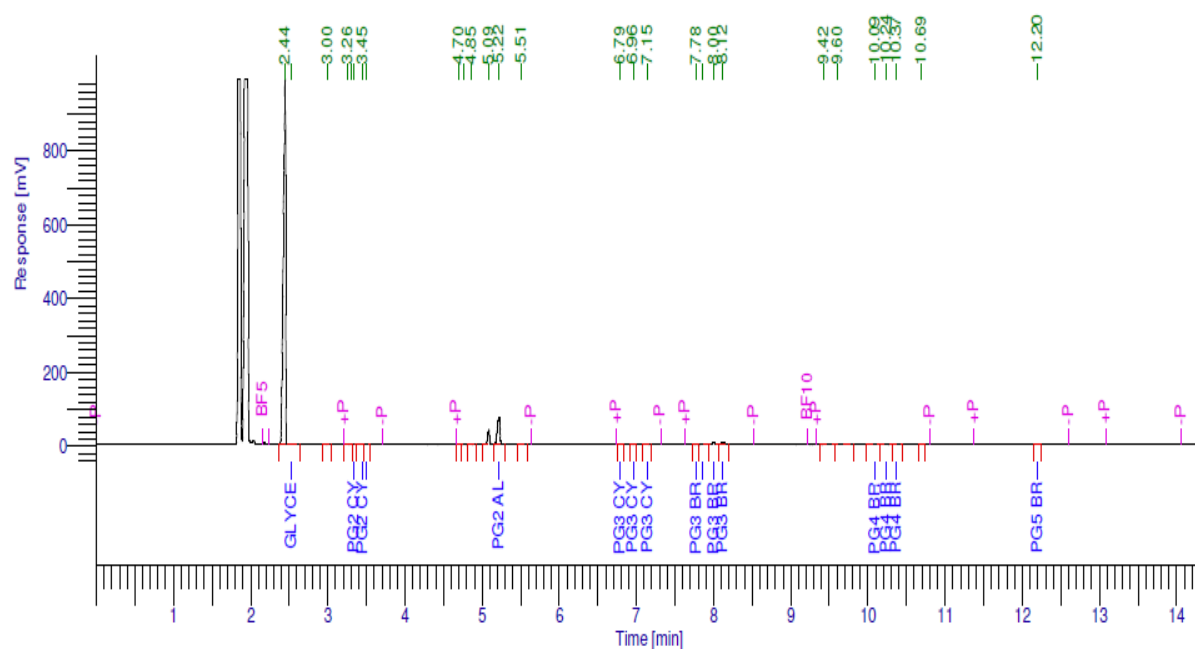
**Figure SI.8:** GC chromatogram of glycerol oligomerization by microwaves at 238 °C at 0.5mL/min continuous flow.



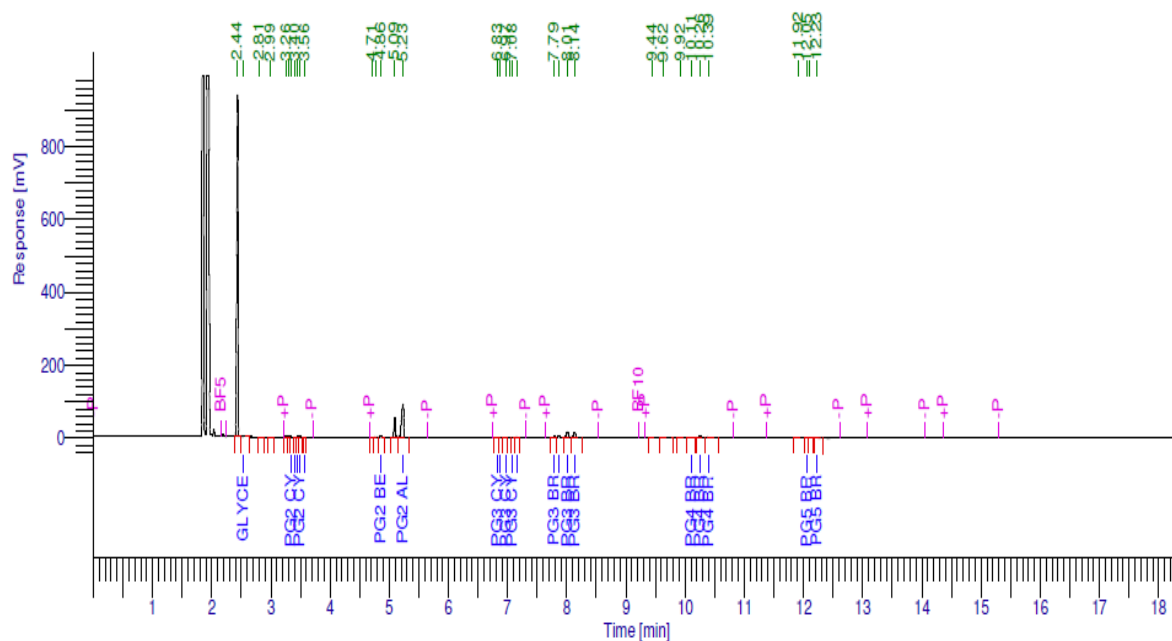
**Figure SI.9:** GC chromatogram of glycerol oligomerization by microwaves at 238 °C at 0.25mL/min continuous flow.



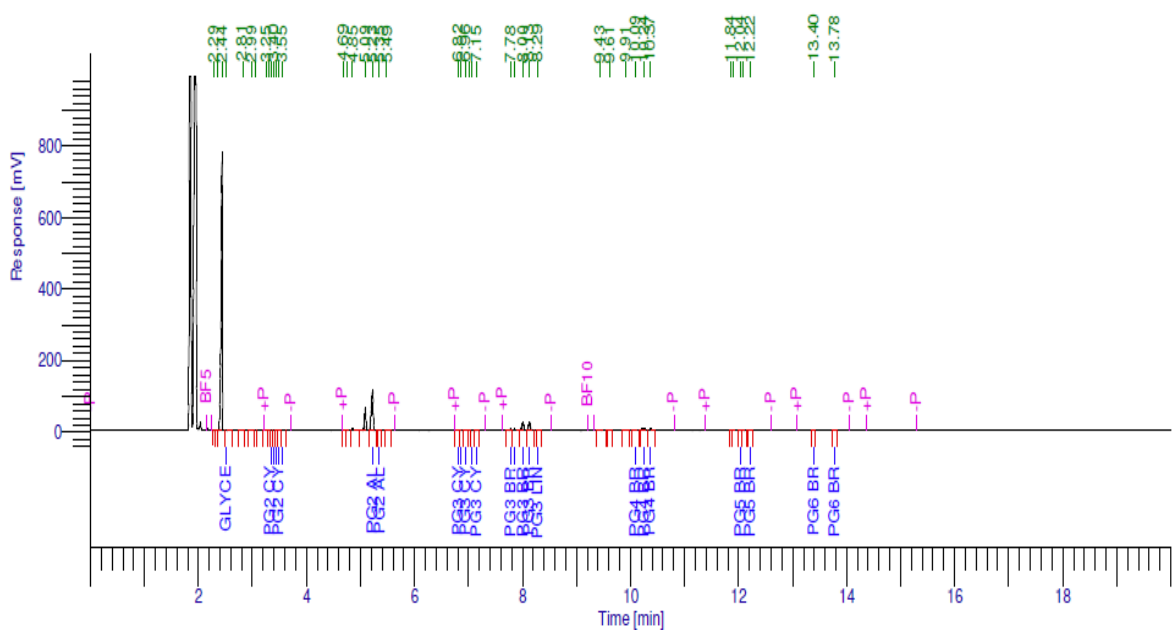
**Figure SI.10:** GC chromatogram of glycerol oligomerization by microwaves at 238 °C at 0.5mL/min continuous flow, injected twice (0.5 ml/min x2)



**Figure SI.11:** GC chromatogram of glycerol oligomerization by microwaves at 238 °C at 0.5mL/min continuous flow in cycle mode after 50 min (cycle 0).



**Figure SI.12:** GC chromatogram of glycerol oligomerization by microwaves at 238 °C at 0.5mL/min continuous flow in cycle mode after 100 min (cycle 1).



**Figure SI.13:** GC chromatogram of glycerol oligomerization by microwaves at 238 °C at 0.5mL/min continuous flow in cycle mode after 150 min (cycle 2).

