

Improved catalyst system for the reduction of levulinic acid to γ -valerolactone

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Electronic Supplementary Information (ESI)

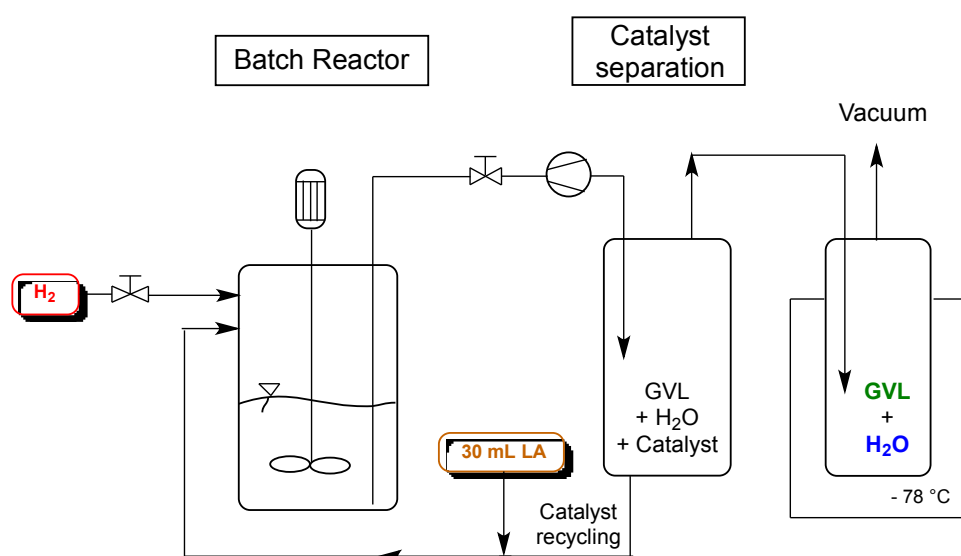


Figure S1. System configuration for catalyst recycling



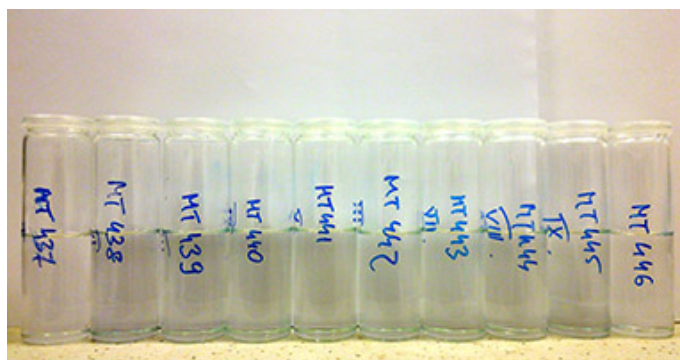
Picture S1: Reaction mixture after complete conversion of LA to GVL



Picture S2: Residue after removal of volatile compounds



Picture S3: Dissolved residue in levulinic acid



Picture S4: 10 distillates

ESI-Table S1. Data for catalyst recycling

Cycle	Catalyst+ligand+ residue (g)*	m (LA) (g)	n (LA) (mmol)	Conversion (%)	m(Distillate) (g)	m(Residue) (g)
1	2.166	33.3	286.8	>99.9	27.64	4.5
2	4.5	32.8	282.5	>99.9	28.02	4.5
3	4.5	33.1	285.1	>99.9	28.59	3.67
4	3.67	32.9	283.4	>99.9	29.06	4.35
5	4.35	33.0	284.2	>99.9	29.07	4.15
6	4.15	33.2	286.0	>99.9	28.42	5.2
7	5.2	33.1	285.1	>99.9	28.93	3.63
8	3.63	32.8	282.5	>99.9	28.66	3.39
9	3.39	32.6	280.8	>99.9	28.8	3.11
10	3.11	32.8	282.5	>99.9	28.2	3.24

* In the initial run: mass of the catalyst+ligand, for the runs 2-10 the mass of the recycled orange glue-like residue containing the catalyst's species.

Sum of LA introduced: 329.6 g

Sum of ten distillates: 285.4 g (86.5 %) due to the efficiency of vacuum distillation (materials remain in the column etc.).

Data for distillation of combined batches:

Sum of distillates: 285.4 g

GVL: 247.5 g

Removed water: 36.3 g

Mass balance: 99.4 %



Picture S5: Levulinic acid obtained by dehydration of D-fructose.

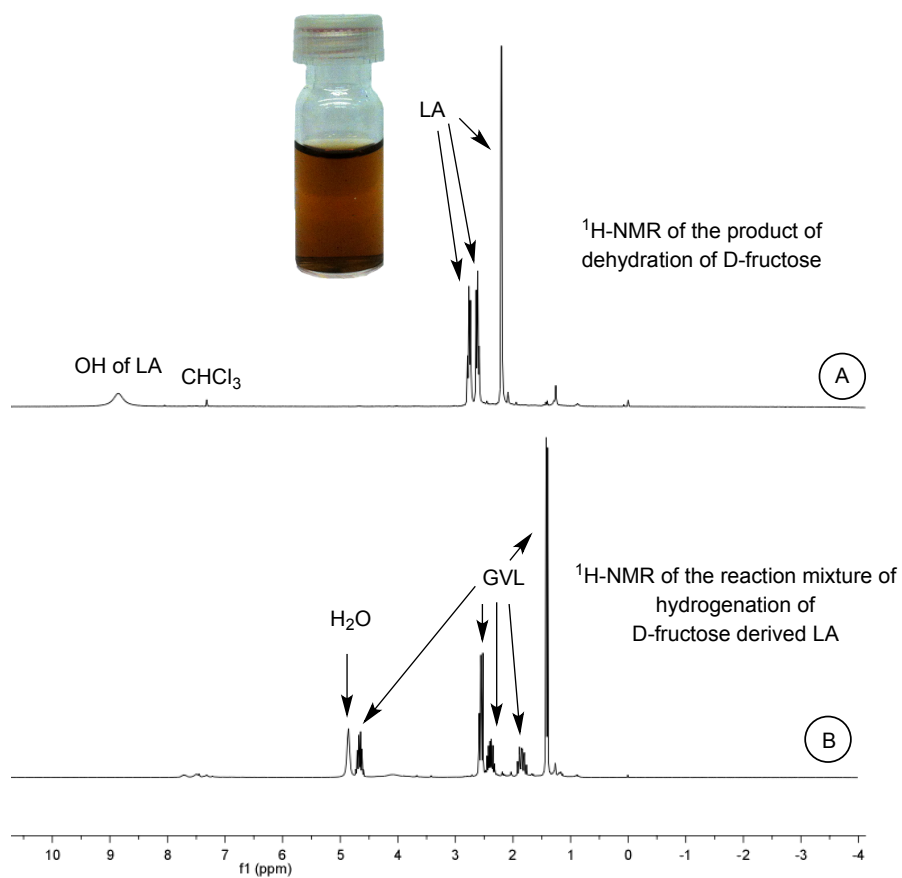


Figure S2. $^1\text{H-NMR}$ spectra of D-fructose derived levulinic acid (A) and the reaction mixture of its catalytic hydrogenation (B)

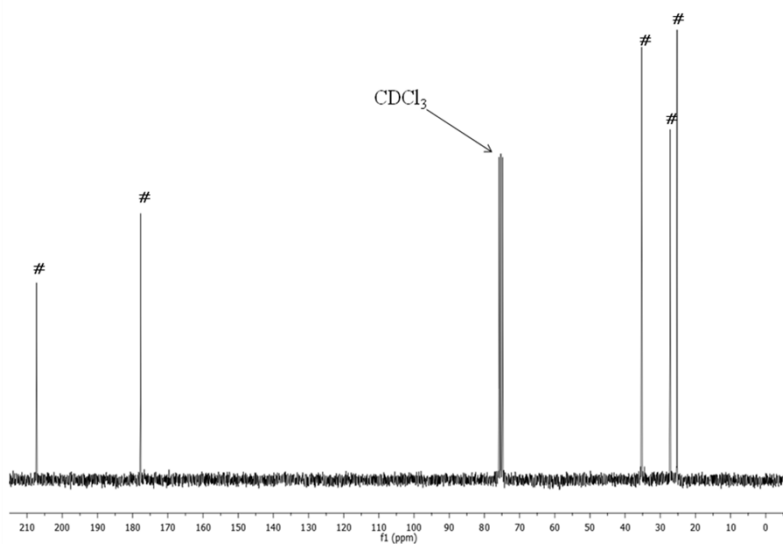


Figure S3. $^{13}\text{C-NMR}$ spectra of D-fructose derived levulinic acid (#).

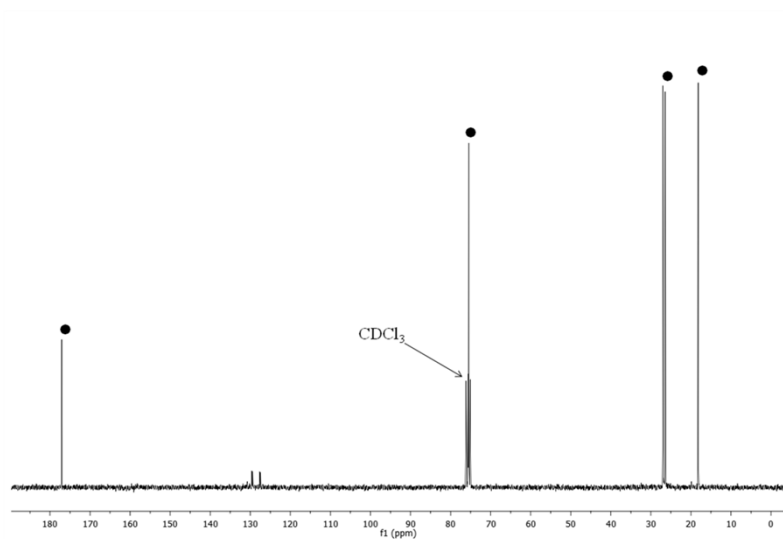


Figure S4. ¹³C-NMR spectra of D-fructose derived levulinic acid reduction to γ -valerolactone (•).