

Highly basic solid catalysts obtained by spray drying of a NaAlO₂ and boehmite suspension for the upgrading of glycerol to acetins

Sreerangappa Ramesh^a Makhlouf Amoura^b and Damien P. Debecker^{a,*}

^a Institute of Condensed Matter and Nanosciences (IMCN), UCLouvain, Place Louis Pasteur,
1, box L4.01.09, 1348 Louvain la-Neuve, Belgium.

^b Dequenne Chimie S.A, Route de Wallonie- Darse Nord, Ghlin, Belgium

* Corresponding author: damien.debecker@uclouvain.be

Supplementary materials

Synthesis of the pure spray-dried sodium aluminate catalyst

The pure NaAlO_2 catalysts was prepared by spray drying (“Type II” aerosol processing), according to our earlier reported procedure.¹ Briefly, 10 ml of sodium aluminate solution (25 wt.%) was diluted in demineralized water (100 ml) and the mixture was sprayed with a 6-Jet 9306A atomizer from TSI with an air pressure of 30 psi and the aerosol was dried by passing through a tubular quartz tube set at 700 °C. The recovered powder was further dried for one night at 120 °C under vacuum and then stored in a desiccator. It is denoted as 100 SA.

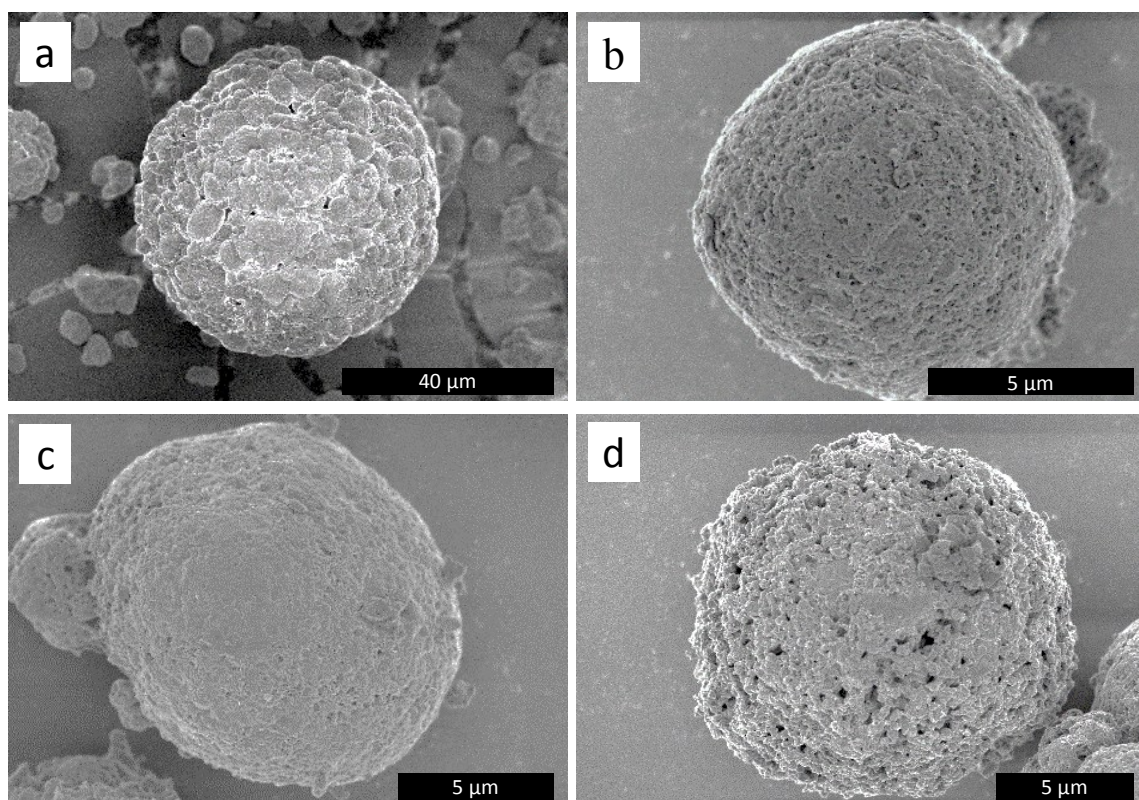


Fig. S1. SEM micrographs of a) boehmite (sample B), b) 5SAB, c) 10SAB and d) 20SAB.

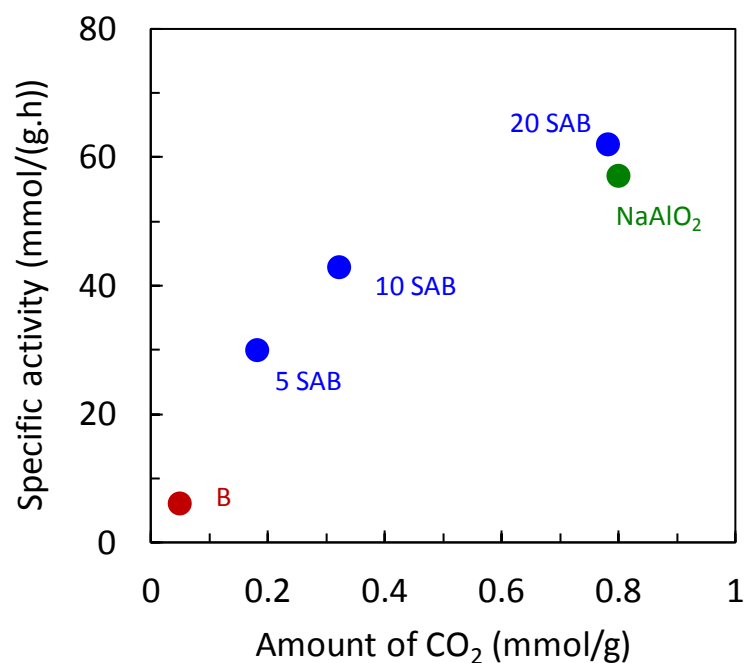


Fig. S2. Correlation between the initial specific activity (approached from the conversion obtained at 1h) and the “basicity” (more precisely, the amount of CO₂ adsorbed and desorbed in the CO₂-TPD experiment). Note that specific activity is probably under-estimated for the most active catalysts, since conversion levels were already relatively high at 1h. Nevertheless, a clear trend can be observed, showing that intrinsic activity increases with CO₂ uptake, itself determined by the NaAlO₂ loading.

Table S1. Conversion of glycerol to glycerol acetins with different catalysts, estimated TOF and carbon

Catalyst	Glycerol conversion (%) ^a	Yield (%)			TOF (h ⁻¹) ^c	Carbon balance ^e
		Mono	Di	Tri		
Blank	4 ^b	4	-	-	-	--
B	6 ^b	6	-	-	-	99
5 SAB	30	21	8	1	167	99
10 SAB	43	28	13	1	134	98
20 SAB	62	35	24	3	80 ^d	98
NaAlO ₂	57	33	21	3	71	98
MgO	37	30	7	0.4	-	99
Hydrotalcites	45	32	12	1	-	99

balance based on glycerol

^aReaction conditions: 0.10 g of catalyst, mole ratio of 1:10 (Gly: MA) at 60°C for 1h

^bSame reaction conditions, but maintained for 16h.

^cTOF is expressed as mol of glycerol converted per mol of “basic sites” and per hour. It is important to note that this is only a rough estimation that should be considered with precaution, because the number of active sites (= basic sites) is only approached by the number of CO₂ detected in TPD. It is based on the conversion level at 1h. Note that TOF is probably underestimated for the most active catalysts because conversion was already relatively high at 1h in those cases.

^dFor SA20, the catalyst appears to be deactivated at 4h of reaction. A TON of 123 can be calculated, based on the same rough approach to the number of active sites.

^eBased on glycerol.

References

1. S. Ramesh and D. P. Debecker, *Catalysis Communications*, 2017, **97**, 102-105.