

Alkali- and nitrate-free synthesis of highly active Mg-Al hydrotalcite coated alumina for FAME production

J. J. Creasey,^a A. Chierigato,^b J.C. Manayil,^c C. M. A. Parlett,^a K. Wilson^c and A. F. Lee^{a,d}

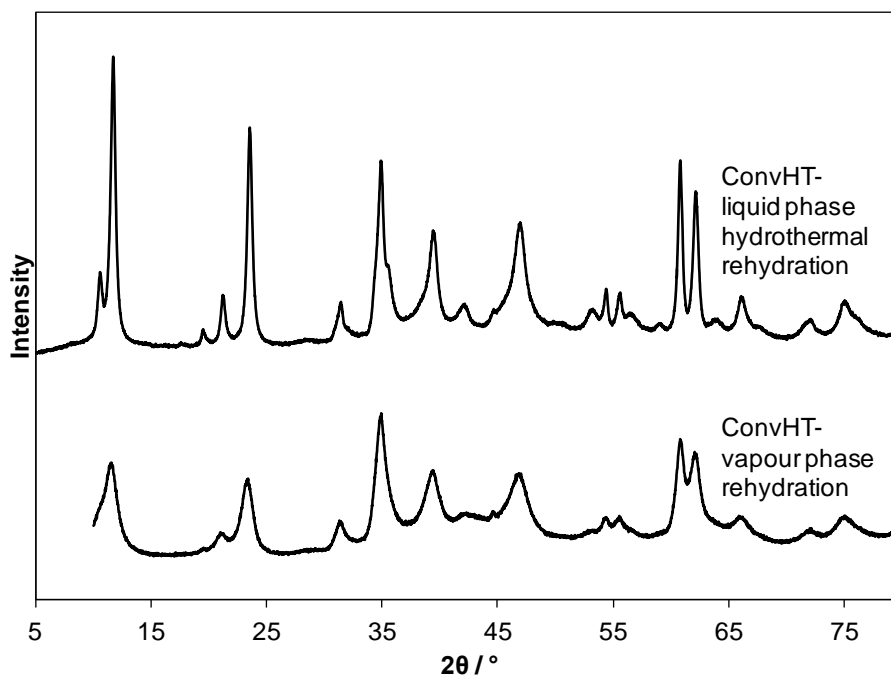


Fig. S1 Powder XRD patterns for conventionally prepared, co-precipitated 2:1 ConvHT reference sample after vapour phase versus hydrothermal liquid phase rehydration. Hydrothermal rehydration enhances hydrotalcite crystallinity

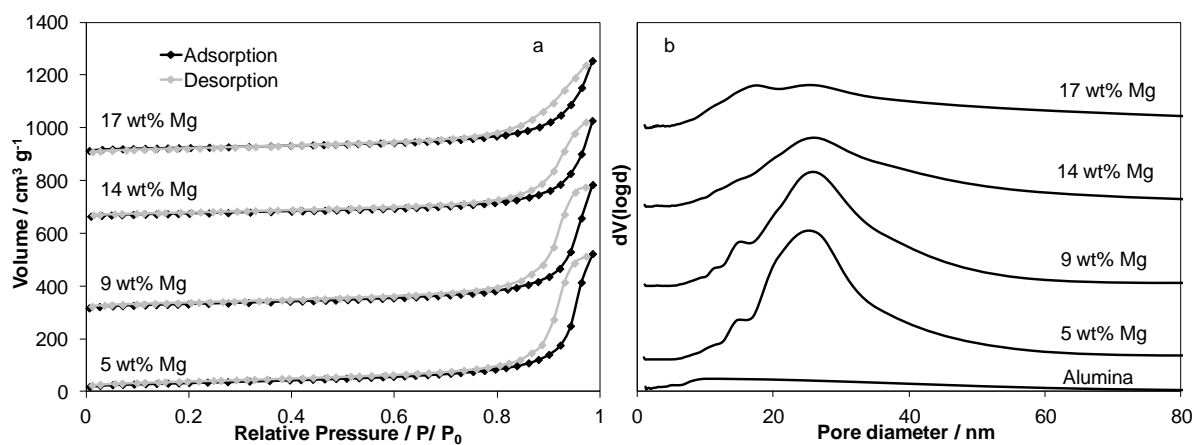


Fig. S2 (a) N_2 adsorption-desorption isotherms; and (b) BJH pore size distributions for Mg-HT/ Al_2O_3 series

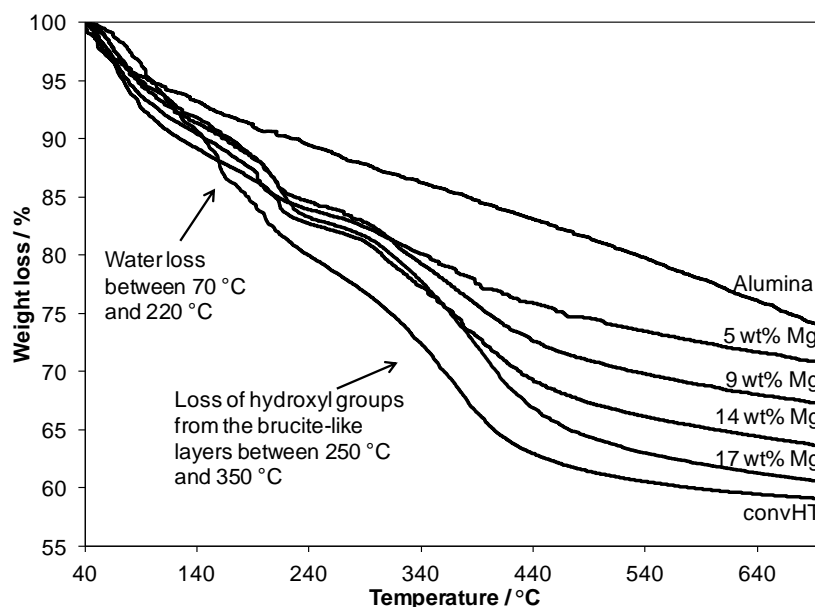


Fig. S3 TGA profiles for the HT/ Al_2O_3 series as well as for 2:1ConvHT reference sample and parent alumina

Table S1 XRD peak intensity ratios for the Mg-HT/ Al_2O_3 series and 2:1ConvHT reference sample

Sample	d(003):d(006) intensity ratio	d(003):d(110) intensity ratio
5 wt% Mg	1.09	0.68
9 wt% Mg	1.17	0.83
14 wt% Mg	1.34	1.17
17 wt% Mg	1.38	1.19
2:1ConvHT	1.09	0.87

Table S2 Maximum surface coverage of HT coating for Mg-HT/ Al_2O_3 series estimated from the initial parent alumina surface area ($110 \text{ m}^2 \cdot \text{g}^{-1}$) and surface density of Mg atoms within Mg_2Al hydroxalcalcite phase (13.5 \AA^2 per Mg atom)

Bulk Mg loading / wt%	5	9	14	17
Mg atoms added in synthesis	9.92×10^{21}	1.98×10^{22}	3.97×10^{22}	4.96×10^{22}
Mg:Al ratio of HT phase	1.79	1.9	2.13	2.08
Mg atoms required for HT monolayer (ML)	3.52×10^{21}	3.73×10^{21}	4.19×10^{21}	4.08×10^{21}
Theoretical maximum HT coverage / ML	2.82	5.30	9.48	12.13
Theoretical maximum HT crystallite size (0.76 x ML) / nm	2.14	4.02	7.20	9.22
Observed HT crystallite size / nm	27	33	36	31
Maximum surface coverage / ML	0.08	0.12	0.20	0.30

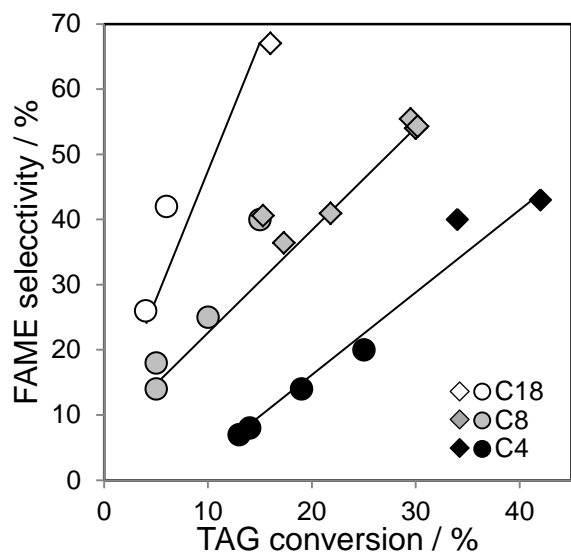


Fig. S4 Linear dependence of FAME selectivity on TAG conversion for Mg-HT/Al₂O₃, conventionally co-precipitated microporous and hierarchical macroporous hydrotalcite catalysts (from *Energy Environ. Sci.* **2012**, *5*, 6145).

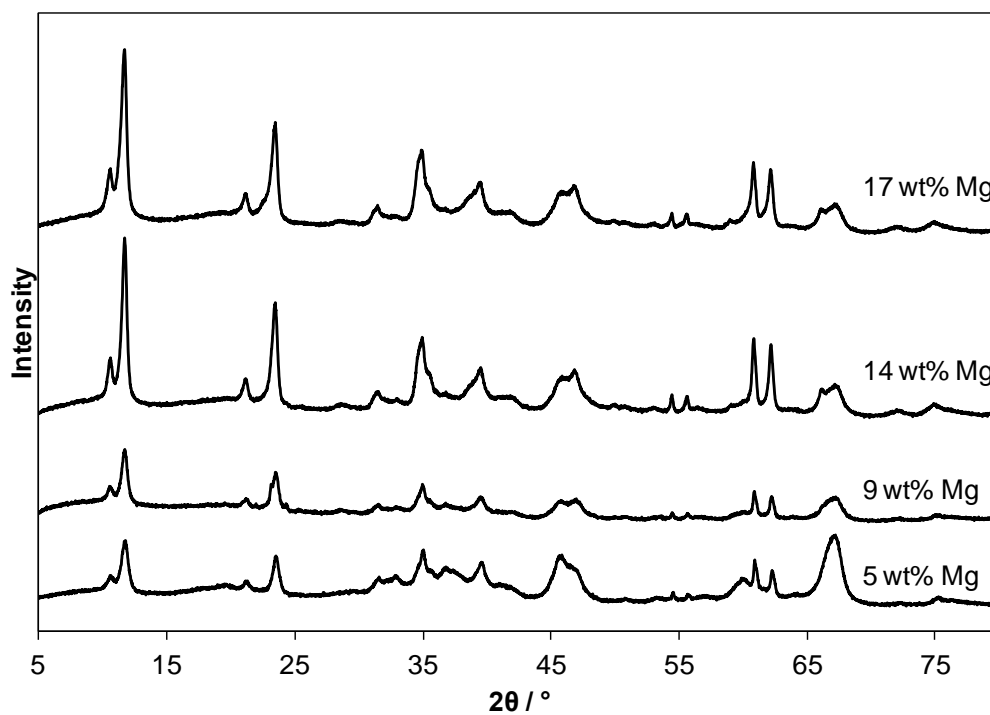


Fig. S5 Powder XRD patterns of spent Mg-HT/Al₂O₃ series following 24 h transesterification of tributyrin with methanol at 60 °C showing retention of hydrotalcite phase.