Supporting Information

Coupling of Solvent-free synthesis and reactive extrusion of alumina: an ecologically efficient integration for heterogeneous catalysts synthesis

AUTHOR NAMES

Pierre-Igor Dassie ^{‡1}, Ryma Haddad ^{‡1}, Maud Lenez¹, Alexandra Chaumonnot², Malika Boualleg², Patrick Legriel¹, Ales Styskalik⁴, Bernard Haye¹, Mohamed Selmann³, Damien P. Debecker⁴, Clement Sanchez¹, Corinne Chaneac¹ and Cedric Boissiere¹*.

AUTHOR ADDRESS

- 1. Laboratoire de Chimie de la Matière Condensée de Paris (LCMCP), Collège de France Sorbonne Université, CNRS, 4 Place Jussieu, 75252 Paris, France.
- 2. IFP Energies Nouvelles (IFPEN), BP 3, F-69360 Solaize, France.
- 3. Fédération de Chimie et Matériaux de Paris-Centre (FCMat) Sorbonne Université, Paris France.
- 4. Institute of Condensed Matter and Nanosciences (IMCN), Université catholique de Louvain (UCLouvain), Place Louis Pasteur, 1, 1348 Louvain-La-Neuve, Belgium).

Scamex's Twin Screws Micro-Extruder (TSME)



SI. 1. A) TSME picture with the doser (1) and the peristaltic pump (2); B) scheme of the experimental set up associated with the picture of screws design; C) Zoom into the extruder head with 0.5 cm orifice size, in the left, and zoom into screw profil: kneading module 1.8×1.8 cm and the transport module 1.8×3.5 cm.

Aluminium isopropoxide XRD diffractogram



SI. 2. XRD pattern of AIP precursor.

Microtome TEM picture



SI. 3. Microtome TEM picture of RE_AIP_8_D sample, presence of some heterogeneous domains of small platelets.

Nitrogen physisorption isotherms of the extrudates



SI. 4. Nitrogen adsorption-desorption isotherms, recorded at 77 K, for samples with hydrolysis rate h= and h=8. The dried extrudates are represented in black and extrudates calcined at 540 °C are represented in red.



SI. 5. Pore size distribution curves for samples with hydrolysis rate h=6 and h=8. The dried extrudates are represented in black and extrudates calcined at 540 °C are represented in red.



Characterization of the extrudates made by the kneading extrusion pathway

SI. 6. Characterization of the extrudates obtained in batch synthesis by sol-gel solvent-free synthesis and shaped in a second time by the usual kneading, peptisation, neutralization, followed by extrusion pathway. Analyses are performed before and after calcination. The obtained extrudates before calcination are labeled with D (D stands for Dry), extrudates after calcination are labeled with C and ASB stands for Aluminum tri-Sec-Butoxide. a) XRD pattern; b) N₂ physisorption isotherms; c) Pore size distribution curves.

Catalytic stability during ethanol dehydration catalytic test



SI. 7. Time-on-stream evolution of ethanol conversion and Ethylene and Diethyl Ether selectivity for the tested samples at 255 $^{\circ}$ C.