Efficient one-pot production of γ-valerolactone from xylose over Zr-Al-Beta zeolite: rational optimization of catalyst synthesis and reaction conditions

Juan A. Melero^{a,*}, Gabriel Morales^a, Jose Iglesias^a, Marta Paniagua^a, Clara López-Aguado^a, Karen Wilson^b, Amin Osatiashtiani^b

Green Chemistry

Electronic Supplementary Material



Figure ESI-1. Reaction network for xylose conversion in 2-propanol into γ-valerolactone through alternative acid catalysed (red) and MPV (blue) reactions.



Figure ESI-2. Proposed reaction scheme for the retro-aldol condensation of xylose into 2propyl lactate and 2-propoxy glycol.



Figure ESI-3. a) Argon adsorption-desorption isotherms, and b) XRD patterns of Zr-Al-Beta zeolites.



Figure ESI-4. DRIFT signal of adsorbed pyridine on (a) Beta, (b) Zr-Al-Beta-1, (c) Zr-Al-Beta-2, (d) Zr-Al-Beta-3, (e) Zr-Al-Beta-4, (f) Zr-Al-Beta-5, (g) Zr-Al-Beta-6, (h) Zr-Al-Beta-7 zeolites.



Figure ESI-5. Xylose conversion and yields to products in 2-propanol over Zr-Al-Beta-6 (Table 1). Reaction conditions: 170° C, xylose:2-propanol molar ratio 1:50, catalyst loading 10 g·L⁻¹.



Figure ESI-6. Fitting goodness representation for the GVL yield model to experimental data. Reaction results at 24h. Error bars refer to the confidence of the model at 95%.



Figure ESI-7. Reaction products distribution as a function of the reaction temperature. Top, 150 °C; Middle, 170 °C; Bottom, 190 °C. Starting xylose concentration = 40 g·L⁻¹; Catalyst loading = 10 g·L⁻¹.



Figure ESI-8. Reaction products distribution as a function of the starting xylose concentration. Top, 10 g·L⁻¹; Middle, 40 g·L⁻¹; Bottom, 70 g·L⁻¹. Reaction temperature = 170°C; Catalyst loading = 10 g·L⁻¹.



Figure ESI-9. Xylose conversion and yields to products in 2-propanol over Zr-Al-Beta-6 (Table 1). Optimized reaction conditions: 190 °C; catalyst loading 15 g·L⁻¹; starting xylose concentration 30.5 g·L⁻¹.



Figure ESI-10. Hot filtration test of Zr-Al-Beta-6 under the optimized reaction conditions. Catalyst was removed after 2h and the reaction was continued without catalyst (red). For comparison purposes, the reaction with catalyst is also included (black). Reaction conditions: 190°C, catalyst loading 15 g·L⁻¹, xylose concentration 30.5 g·L⁻¹.