Synthesis of Mesoporous Metal Complex-Silica Materials and their Use as Solvent-free Catalysts

Noemi Linares¹, Angel E. Sepúlveda², María C. Pacheco³, Jesús R. Berenguer^{2,*}, Elena Lalinde², Carmen Nájera³, Javier Garcia-Martinez^{1,*}

¹ Laboratorio de Nanotecnología Molecular. Departamento de Química Inorgánica. Universidad de Alicante, Carretera San Vicente s/n, E-03690, Alicante, Spain. e-mail: <u>j.garcia@ua.es</u> URL: <u>www.ua.es/grupo/nanolab</u>

² Departamento de Química-Grupo de Síntesis Química de La Rioja, UA-CSIC. Universidad de La Rioja, E-26006, Logroño, Spain. URL: <u>www.unirioja.es/dptos/dq/grupos/materiales</u>

³ Grupo de Procesos Catalíticos en Síntesis Orgánica. Departamento de Química Orgánica. Universidad de Alicante, Carretera San Vicente s/n, E-03690, Alicante, Spain.

Supporting information



Figure S1. (a) N₂ isotherms for catalyst MSU_PdCl₂(PETS)₂ prepared with 1%Pd:SiO₂ with (Δ -) and without (- \blacktriangle -) ethanol in their synthesis. (b) Representative TEM images of these samples prepared with (left) and without (right) ethanol in the synthesis. The scale bars represent 10 nm

Supplementary Material (ESI) for New Journal of Chemistry # This journal is (c) The Royal Society of Chemistry and The Centre National de la Recherche Scientifique, 2010



Figure S2. DRUV spectra of (a) pressed pure powders of the MSU-PdCl₂(APTS)₂ material (solid line) and the monomer complex [trans-PdCl₂(APTS)₂] 1 (dotted line) and (b) pressed pure powders of the MSU-PdCl₂(PPETS)₂ material (solid line) and the monomer complex [trans-PdCl₂(PPETS)₂] 3 (dotted line).

Supplementary Material (ESI) for New Journal of Chemistry # This journal is (c) The Royal Society of Chemistry and The Centre National de la Recherche Scientifique, 2010



Figure S3. FT-IR spectra of (**a**) the MSU-PdCl₂(APTS)₂ silica material (up) and the monomer complex [trans-PdCl₂(APTS)₂] **1** (down) and (**b**) the MSU-PdCl₂(PPETS)₂ silica material (up) and the monomer complex [trans-PdCl₂(PPETS)₂] **3** (down). For clarity, two magnifications or the marked areas in the spectra are shown in the left.

Supplementary Material (ESI) for New Journal of Chemistry # This journal is (c) The Royal Society of Chemistry and The Centre National de la Recherche Scientifique, 2010



Figure S4. SEM images of the (a) MSU_PdCl₂(APTS)₂ material prepared from amine ligand and 0.65 wt% loading of Pd, (b) MSU_PdCl₂(PETS)₂ material prepared from pyridine ligand and 0.75 wt% loading of Pd and (c) MSU_PdCl₂(PPETS)₂ material prepared from phosphine ligand and 0.70 wt% loading of Pd. (d) X-ray elemental mapping images for a representative sample of MSU_PdCl₂(PETS)₂ (0.75 wt% Pd:SiO₂), blue, green and red dots represent silicon, oxygen and palladium atoms, respectively.