Electronic Supplementary Information for

Boronate self-assembles with embedded Au nanoparticles; preparation, characterization and their catalytic activities for the reduction of nitroaromatic compounds

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Synthesis of Au/SiO₂ hybrids

Au/SiO₂ catalysts were prepared according to the procedure reported by Corma et al.1 10 mL of ethanol solution containing the dispersed capped Au NPs were added to a solution formed by 1.85 mL of TEOS (tetra-ethyl-ortho-silicate) and 0.25 mL of H₂O. The hydrolysis was catalysed by 0.6 mL of an aqueous solution of NH₄F (0.045 M). The sol under continuous stirring became in tens of minutes a gel that slowly converted in a white, slightly yellow powder. The powder was finally dried overnight at 450 °C. In this way, 474 mg of Au/SiO₂ catalysts was obtained. The amount of loaded Au was 1.6 wt%, determined by ICP-AES. For the characterization, the TEM micrograph and histogram of the particle size distribution are shown in Fig. S1.


Fig. S1 (a) The TEM image and (b) the particle size distribution of Au/SiO₂.

Fig. S2 The size distribution of boronate microparticles BP.
**Fig. S3** FE-SEM image of boronate ester polymers prepared from **1** \((5.0 \times 10^{-2} \text{ mmol})\) and **2** \((5.0 \times 10^{-2} \text{ mmol})\) in DMSO (5 mL) at room temperature for 5h.

**Fig. S4** Thermogravigram (TG) curve of **BP**. The graph shows the loss of mass as a function of temperature. Conditions: nitrogen atmosphere and heating rate of 5°C min\(^{-1}\) in the temperature range of 22.7–878.2°C.
**Fig. S5** HR-TEM image of Au NP deposited on Au–BP(SG) by solid griding (SG) method.

**Fig. S6** (a) UV/vis absorption spectra for the reduction of 4-NP with NaBH₄ over Au–BP(SG) in methanol at 25 °C. The base line was normalized due to a slight precipitation during the reaction. (b) Plot of ln(Aₜ/A₀) versus time for the reduction of 4-NP. Reaction condition, [Au] = 7.1 × 10⁻⁵ M, [4-NP] = 1.3 × 10⁻⁴ M, and [NaBH₄] = 1.8 × 10⁻² M.
Fig. S7 FE-SEM image of Au-BP(SG).

Fig. S8 (a) Size distribution of Au NPs, being based on TEM image, for Au–BP(DR) after the second run for the hydrogenation. The average diameter was determined to be 2.8 ± 0.6 nm. (b) FE-SEM image for Au–BP(DR) after the second run for the hydrogenation.

Fig. S9 FE-SEM image of Au-BP(DR) after five successive runs for reduction of 4-NP with NaBH₄ in methanol.