

Supporting Information

Scalable Synthesis of Catalysts for the Mizoroki-Heck Cross Coupling Reaction: Palladium Nanoparticles Assembled in a Polymeric Nanosphere

Jianli Zou,^a K. Swaminathan Iyer^{*a}, Scott G. Stewart^{*b} and Colin L. Raston^a

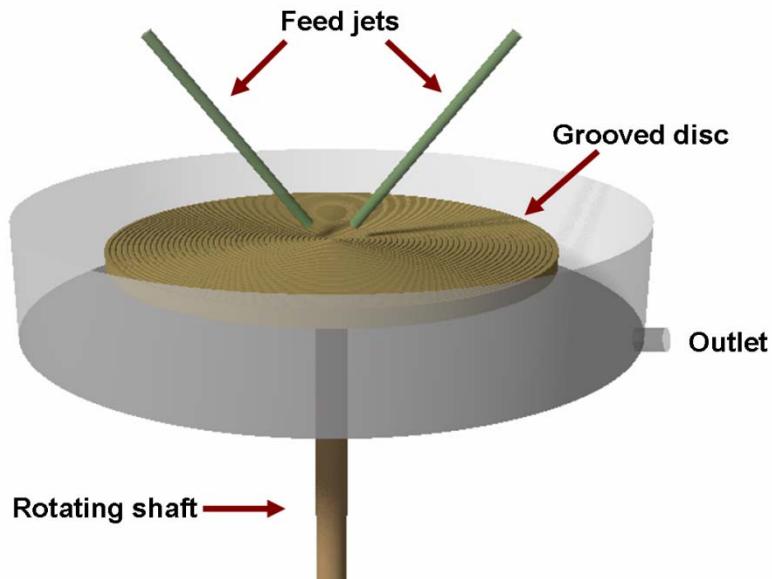


Figure S1. Schematic of Spinning Disc Processor (SDP)

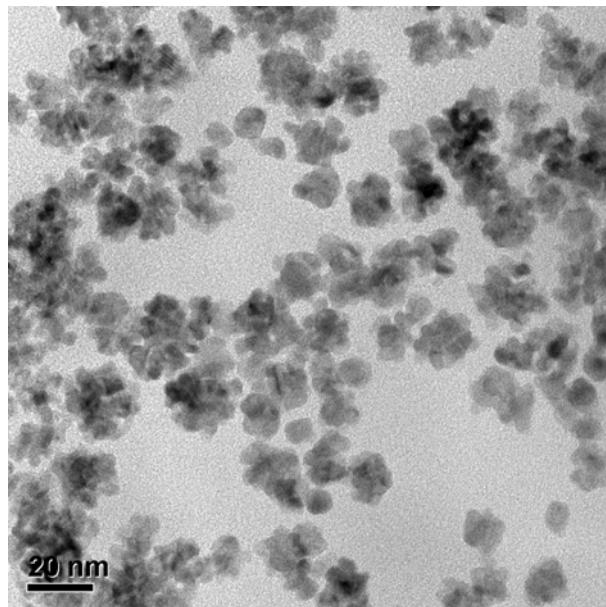


Figure S2. TEM image of as-prepared palladium nanoparticles using hydrazine as reducing reagent (other parameters are same as nano-spheres showing in Figure 1(a)).

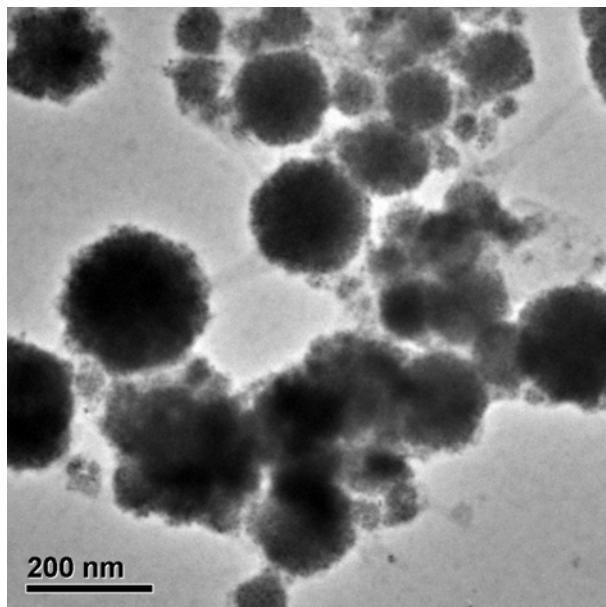


Figure S3. TEM image of a typical palladium nanomaterials synthesized using PVP with a molecular weight of 360,000 with a disc speed of 2500 rpm.

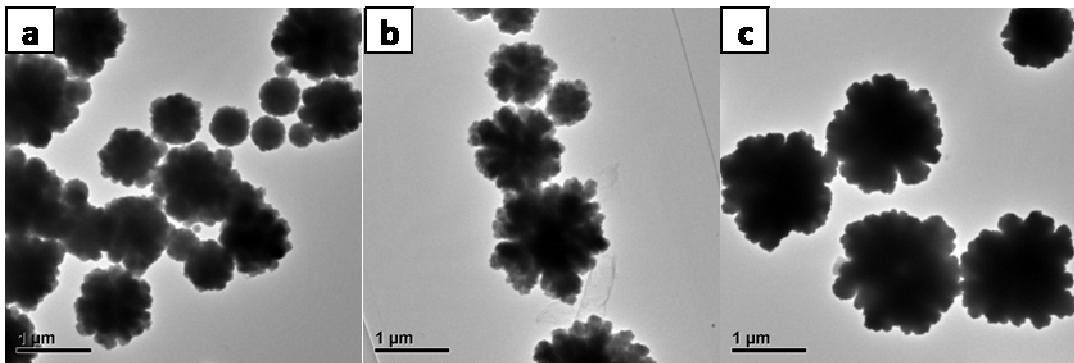


Figure S4. TEM images of palladium-PVP spheres synthesized via mechanical stirring by using (a) PVP10 (MW 10,000); (b) PVP40 (MW 40,000) and (c) PVP360 (MW 360,000).

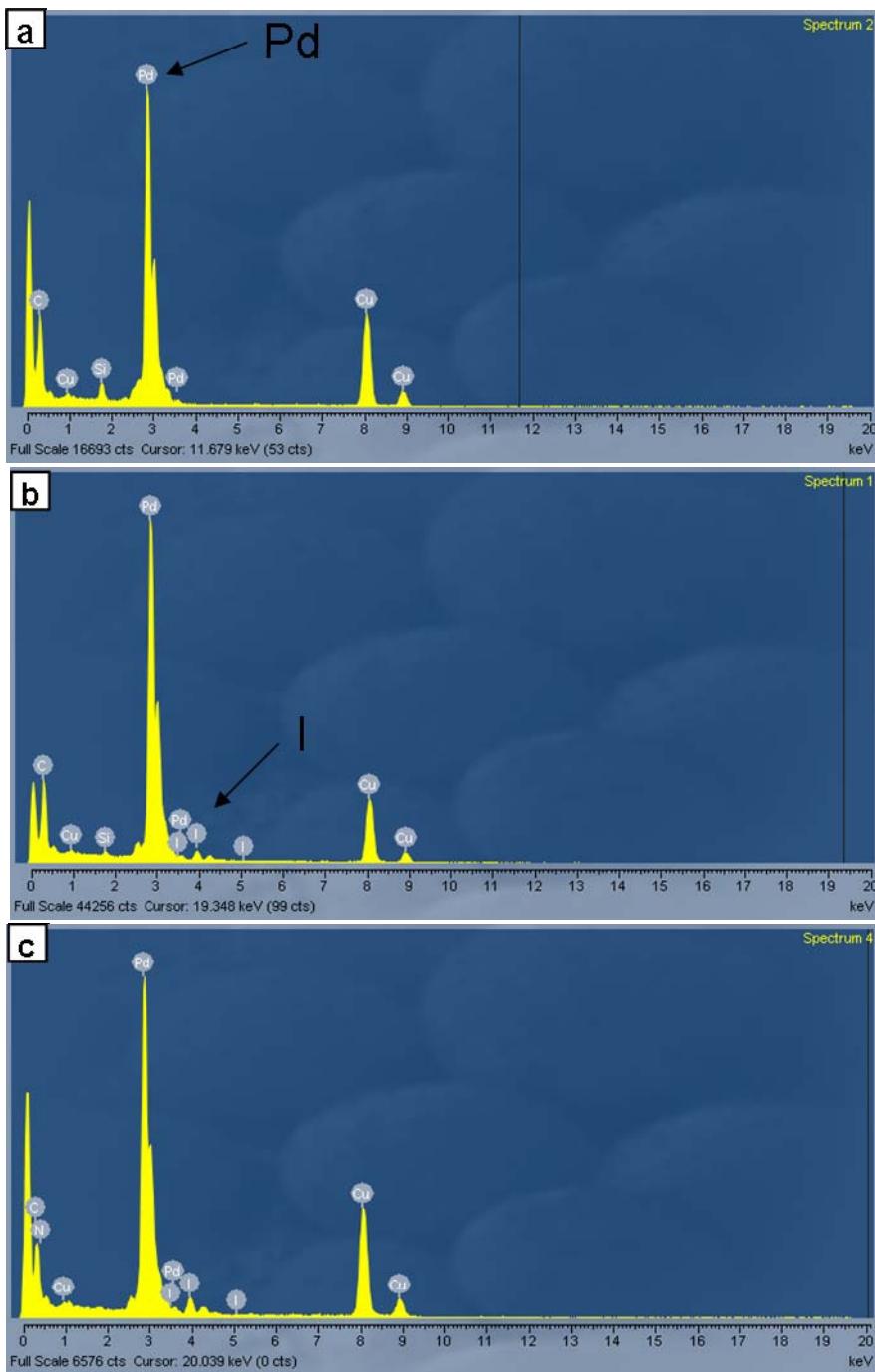
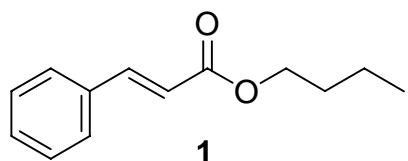
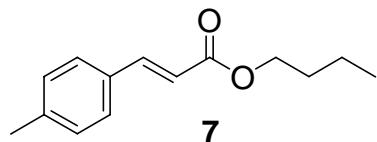


Figure S5. EDS data of palladium nanospheres. (a) pristine; (b) after 1st run; (c) after 5th run.

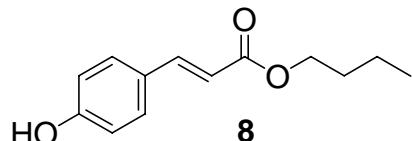
¹H NMR data and GC-MS data



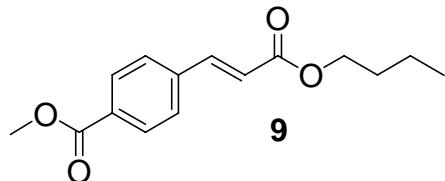
¹H NMR (400 MHz, CDCl₃, ppm): δ 7.68 (d, 1H, *J*=16.0 Hz), 7.54–7.52 (m, 2H), 7.39–7.37 (m, 3H), 6.44 (d, 1H, *J*=16.0 Hz), 4.21 (t, 2H, *J*=6.6 Hz), 1.69 (quint, 2H, *J*=7.2 Hz), 1.44 (sextet, 2H, *J*=7.4 Hz), 0.97 (t, 3H, *J*=7.6 Hz); GC-MS *m/z* (relative intensity): 204 (M⁺, 18), 148 (74), 131 (100), 103 (57), 77 (38).



¹H NMR (400 MHz, CDCl₃, ppm): δ 7.63 (d, 1H, *J*=16.0 Hz), 7.41 (d, 2H, *J*=7.8 Hz), 7.19 (d, 2H, *J*=7.8 Hz), 6.40 (d, 1H, *J*=16.0 Hz), 4.20 (t, 2H, *J*=6.4 Hz), 2.38 (s, 3H), 1.64 (quint, 2H, *J*=7.2 Hz), 1.42 (sextet, 2H, *J*=7.4 Hz), 0.98 (t, 3H, *J*=7.6 Hz); GC-MS *m/z* (relative intensity): 218 (M⁺, 29), 162 (95), 145 (100), 115 (53), 91 (26).

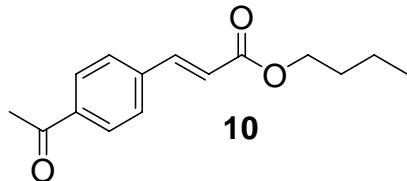


¹H NMR (400 MHz, CDCl₃, ppm): δ 7.63 (d, 1H, *J*=16.0 Hz), 7.41 (d, 2H, *J*=8.4 Hz), 6.87 (d, 2H, *J*=8.4 Hz), 6.30 (d, 1H, *J*=16.0 Hz), 5.15 (s, 1H), 4.22 (t, 2H, *J*=6.4 Hz), 1.68 (quint, 2H, *J*=5.6 Hz), 1.43 (sextet, 2H, *J*=7.6 Hz), 0.97 (t, 3H, *J*=7.2 Hz); GC-MS *m/z* (relative intensity): 220 (M⁺, 21), 164 (100), 147 (50), 119 (24), 107 (22).

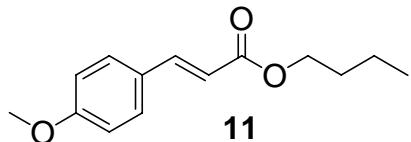


¹H NMR (400 MHz, CDCl₃, ppm): δ 8.03 (d, 2H, *J*=4.0 Hz), 7.67 (d, 1H, *J*=8.0 Hz), 7.56 (d, 2H, *J*=4.0 Hz), 6.50 (d, 1H, *J*=8.0 Hz), 4.21 (d, 2H, *J*=6.4 Hz), 3.91 (s, 3H), 1.68

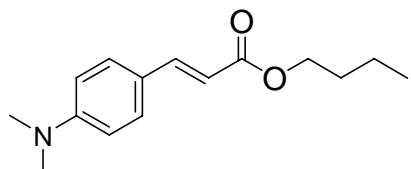
(quint, 2H, $J=7.2\text{Hz}$), 1.42 (sextet, 2H, $J=7.6\text{Hz}$), 0.95 (t, 3H, $J=7.2\text{ Hz}$); GC-MS m/z (relative intensity): 262 (M^+ , 28), 206 (100), 189 (46), 175 (75), 145 (43).



^1H NMR (400MHz, CDCl_3 , ppm): δ 7.97 (d, 2H, $J=4.4\text{Hz}$), 7.69 (d, 1H, $J=8.0\text{Hz}$), 7.61 (d, 2H, $J=4.4\text{Hz}$), 6.53 (d, 1H, $J=8.0\text{Hz}$), 4.23 (t, 2H, $J=6.8\text{Hz}$), 2.62 (s, 3H), 1.70 (quint, 2H, $J=7.2\text{Hz}$), 1.44 (sextet, 2H, $J=7.6\text{Hz}$), 0.97 (t, 3H, $J=7.6\text{Hz}$); GC-MS m/z (relative intensity): 246 (M^+ , 18), 231(46), 190 (39), 175 (100), 131 (32).



^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.67 (d, 1H, $J=8.0\text{ Hz}$), 7.48 (d, 2H, $J=4.4\text{Hz}$), 6.90 (d, 2H, $J=4.4\text{ Hz}$), 6.31 (d, 1H, $J=8.0\text{Hz}$), 4.20 (t, 2H, $J=6.4\text{ Hz}$), 3.84 (s, 3H), 1.69 (quint, 2H, $J=7.2\text{Hz}$), 1.47(sextet, 2H, $J=7.2\text{Hz}$), 0.96 (t, 3H, $J=7.2\text{ Hz}$); GC-MS m/z (relative intensity): 234 (M^+ , 42), 178 (100), 161 (98), 134 (41), 121(31).



^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.61 (d, 1H, $J=16.0\text{ Hz}$), 7.41 (d, 2H, $J=6.8\text{ Hz}$), 6.63 (d, 2H, $J=6.8\text{ Hz}$), 6.22 (d, 1H, $J=16.0\text{ Hz}$), 4.18 (t, 2H, $J=6.8\text{ Hz}$), 3.00 (s, 6H), 1.70-1.64 (m, 2H), 1.46-1.41 (m, 2H), 0.96 (t, 3H, $J=7.2\text{ Hz}$); GC-MS m/z (relative intensity): 247 (M^+ , 94), 191 (41), 174 (62), 147 (100).