Preparation and Characterization of Pd modified TiO_2 nanofiber catalyst for carbon–carbon coupling Heck reaction

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TiO_2 fibers were prepared through electrospinning of polymethyl methacrylate (PMMA) and Titanium isopropoxide (TIP) solution followed by calcination of fibers in air at 500 ºC. CTAB protected Palladium nanoparticles prepared through reduction method were successfully adsorbed on the TiO_2 nanofibers. Combined studies of X-ray diffraction (XRD), Scanning electron microscope (SEM) and Transmission electron microscope (TEM), indicated that the synthesized Pd/TiO_2 was anatase phase. BET indicated that the synthesized TiO_2 and Pd/TiO_2 had a surface area of 53.4672 and 43.4 m^2/g, respectively. The activity and selectivity of 1 mol % Pd $/TiO_2$ in the Heck reaction has been investigated towards the Mizoroki-Heck carbon-carbon cross coupling of bromobenzene and styrene. Temperature, time, solvent and base were optimized and catalyst recycled twice. ^1H NMR and ^{13}C NMR indicated that stilbene, a known compound from literature was obtained in various Heck reactions at temperatures between 100 ºC and 140 ºC. but the recyclability was limited due to some palladium leaching and catalyst poisoning which probably arose from some residual carbon from the polymer. The catalyst was found to be highly active under air atmosphere with reaction temperatures up to 140 ºC. Optimized reaction condition resulted into 89.7 % conversions with a TON of 1993.4 and TOF value of 332.2 hr^{-1}.

Keywords: Heck reaction; calcination; electrospinning; Pd/TiO_2