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Synthesis of Vic-Dioxime Palladium (II) Complex: Precursor for Deposition on SBA-15 in ScCO2

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Abstract : Synthesizing supercritical carbon dioxide ($scCO_2$) soluble precursors would be helpful for many processes of material syntheses based on $scCO_2$. Ligand (a*mphi*-(1*Z*, 2Z)-*N*-(2-fluoro-3-(trifluoromethyl) phenyl)-*N*'-hydroxy-2-(hydroxyimino) were synthesized from chloro glyoxime and flourus aniline and Pd(II) complex (precursor) prepared. For $scCO_2$ deposition method, organometallic precursor was dissolved in $scCO_2$ and impregnated onto the SBA-15 at 90 °C and 3000 psi. Then the organometallic precursor was reduced with H_2 in the CO_2 mixture (150 psi H_2 + 2850 psi CO_2). Pd deposited support material was characterized by ICP-OES, XRD, FE-SEM, TEM and EDX analyses. The Pd loading of the prepared catalyst, measured by ICP-OES showed a value of about 1.64% mol/g Pd of catalyst. Average particle size was found 5.3 nm. The catalytic activity of prepared catalyst was investigated over Suzuki-Miyaura C-C coupling reaction in different solvent with K_2CO_3 at 50 °C. The conversion ratio was determined by gas chromatography.

Keywords : nanoparticle, nanotube, oximes, precursor, supercritical CO2
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