Silica gel-supported Pd nanocatalyst: Efficient Mizoroki-Heck reactions and sustainable Ozagrel synthesis

ABSTRACT

We developed a cost-effective silica gel-supported palladium nanocatalyst in a three-step reactions process. Initially, silica gel (60–120 mesh) underwent amino group functionalization using 3- aminopropyltriethoxysilane, leading to the formation of a Schiff base through a reaction with the 1,10- phenanthroline-2,9-dicarboxaldehyde ligand. Subsequently, palladium nanocatalyst was introduced to the silica matrix ligand in the presence of palladium salt and hydrazine hydrate, resulting in the formation of the silica gel-supported Schiff-base palladium nanocatalyst (Si@SBPdNPs 3). Successful functionalization of the silica matrix was confirmed using various spectroscopic techniques. FT-IR spectra demonstrated the incorporation of organic moieties onto the silica surface, while SEM images revealed the modified spherical shape of the silica gel. TEM and XRD analyses confirmed the presence of palladium on the silica matrix. ICP and EDX measurements validated the anchoring of 0.55 mmol/g of palladium to the catalyst. Additionally, XPS analysis showed the complexation of Pd(0) with the organic ligand on the silica matrix, confirming the successful integration of palladium into the system. This nanocatalyst demonstrated outstanding performance in Mizoroki-Heck reactions, yielding high product outputs in the cross-coupling of various aryl halides and olefins under mild conditions. Additionally, the nanocatalyst was effectively utilized in synthesizing Ozagrel, a thromboxane A2 synthesis inhibitor used for treating noncardioembolic stroke patients. Remarkably, the catalyst demonstrated excellent reusability, maintaining high productivity across five consecutive cycles, underscoring its economic and sustainable potential for industrial applications.