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Synthesis of Ni–P–B amorphous nanoparticles with uniform size as a potential hydrodeoxygenation catalyst

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Electronic supplementary information (ESI)

Catalyst characterization

The surface composition and surface electronic state were analyzed by X-ray Photoelectron Spectroscopy (XPS) using Kratos Axis Ultra DLD instrument at 160eV pass energy. Al K α radiation was used to excited photoelectrons. The surface composition of each sample was calculated according to the corresponding peak areas of Ni, P and B and the sensitivity factors for these elements. X-ray diffraction (XRD) test was carried on a D/max 2550 18KW Rotating anode X-Ray Diffractometer with Cu K α ($\lambda=1.5418\text{\AA}$) radiation (40kV, 300mA). The 2θ was scanned over the range of 10–80° at a rate of 10°/min to identify the amorphous structure. The scanning electron microscope (SEM) images of the catalysts were obtained on a JEOL JSM-6360 electron microscopy. Specific surface area was measured by a Quantachrome's NOVA–2100e Surface Area instrument by physisorption of nitrogen at 77 K. The samples were dehydrated using vacuum degassing for 12 h before experiments.

Catalyst activity measurement

The HDO activity measurements were carried out in a 300–mL sealed autoclave. Fresh catalyst (0.2 g), *p*-cresol (13.51 g) and dodecane (86.49 g) were placed into the autoclave. Air in the autoclave was evacuated by pressurization–depressurization cycles with nitrogen and subsequently with hydrogen. The mixture was heated at 10 K/min to 498 K, then pressurized with hydrogen to 4.0 MPa, stabilized the stirring speed at 700 rpm. During the reaction, liquid samples were withdrawn from the reactor and identified by Agilent 6890/5973N GC–MS. The amounts of *p*-cresol and products were analyzed by Agilent 7890 gas chromatography using a flame ionization detector (FID) with a 30 m AT–5 capillary column. Duplicate or triplicate experiments were performed and the average of these tests is reported here. The errors for conversion values were typically within plus/minus 5.0 mol%. Conversion = (the amount of aromatic-ring change during reaction/total amount of aromatic-ring) \times 100 %; Selectivity = (C atom in each product/total C atom in the products) \times 100 %; Deoxygenation degree (DD, wt %) is defined as [1-oxygen content in the final organic compounds / total oxygen content in the initial material] \times 100%. Carbon balance is better than 96 \pm 3 % in this work.