

Preparation and Characterization of Mesoporous Polymer-Based Solid Acid Catalysts for Biodiesel Production Enhancement

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The global warming has gained increased concerns, and the technology of international energy trends has been significant changed. It is estimated that fossil energy supplied will be still relied in 2030, and threats to the security of global energy. It is an emergent bioenergy for which it is important to guarantee environmental sustainability. Current liquid biofuel production processes rely on first-generation conversion pathways and comprise two distinct products: bioethanol and biodiesel. In this work, the objectives were to synthesize complete solid acidic catalyst PDVB-SO₃H with different solvents in optimal conditions. In addition, this research also investigated the efficiencies of esterification and transesterification. Experimentally, several experimental techniques were used; such as XRD, TGA, FE-SEM, HR-TEM, N₂ adsorption, GC-MS, FTIR, ESCA, Contact angle meter, Raman, NMR and XAS (XANES/EXAFS). The FE-SEM images of the solid acid synthesized with acetonitrile showed that the resulting products were sphere structures with smooth surface. The particle sizes of PDVB in this work were about 1-2 μm. Further increasing the sulfonate group amount, the PDVB particles surfaces would cover with sulfonate group and the resin-like structure was formed instead. Solid acid catalyst synthesized with ethyl acetate showed unpolished resin-like structure. TG curves show a weight loss at 240-380, and 470-590°C, which are assigned to decomposition of sulfonic groups, and destruction of the polymer network, respectively. BET surface area, N₂ isotherms and the corresponding pore size distribution of PDVB-x-SO₃H synthesized with acetonitrile and ethyl acetate with molar ratios of sodium p-styrene sulfonate to DVB. Clearly, all the samples showed typical type-IV isotherms, giving a steep increase at a relative pressure of $0.6 < P/P_0 < 0.9$, indicating the presence of mesoporosity. FT-IR spectra of PDVB-x-MeCN and PDVB-x-EAC samples at 1010, 1035, 1125, and 1220 cm⁻¹. Notably, the band at around 1035 cm⁻¹ is associated with the presence of a C—S bond on the benzene rings, and the bands at around 1010, 1125, and 1220 cm⁻¹ are associated with the asymmetric and symmetric stretching signals of the O=S=O bonds of a sulfonic group. The NMR resonance peaks at 113.9 and at 143.75 ppm are attributed to the carbon atoms, =CH₂ and C—SO₃, respectively. The aromatic carbons in the ortho- or meta-position of the SO₃ group are lumped together in a broad peak centered at 125.8 ppm, XPS measurements of solid acid catalyst, which exhibits the obvious signals of C, S, and O elements. The S 2p and S 2s peaks at 169.1 and 233 eV are assigned to S—O and S=O bonds. The C 1s peaks around 284.8 and 286.1 eV are associated with C—C and C—S bonds, confirming the sulfonic groups functionalized on the polymer network, in good agreement with results obtained from FT-IR spectroscopy.

Keywords: Biodiesel, Esterification, Polymer-Based, Solid acidic catalysts, Sulfonation, Free fatty acid.

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