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Preparation of MCM-41 supported $(Bu_4N)_4H_3(PW_{11}O_{39})$ catalyst and its performance in oxidative desulfurization

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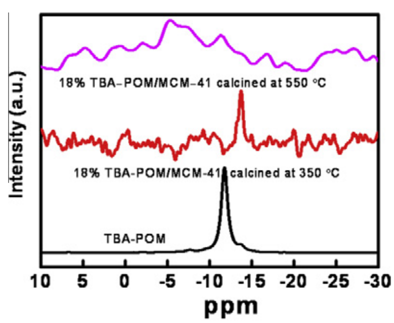
HIGHLIGHTS

- ▶ We prepared and designed TBA–POM/MCM-41 mesoporous material.
- ▶ The characterization results revealed that $(PW_{11}O_{39})^{7-}$ was grafted on MCM-41.
- ▶ The material was found to be highly efficient for catalytic oxidative desulfurization for model and actual oils.

GRAPHICAL ABSTRACT



^{31}P CP-MAS NMR of Samples

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ABSTRACT

Highly ordered mesoporous material $(Bu_4N)_4H_3(PW_{11}O_{39})$/MCM-41 with 10, 18, and 25 wt.% $(Bu_4N)_4H_3(PW_{11}O_{39})$ loadings was prepared by impregnation method in acidic media under reflux conditions, and characterized by XRD, HRTEM, Nitrogen adsorption/desorption, FT–IR, ^{31}P CP–MAS NMR, XPS, SEM and TEM. The characterization results revealed that $(Bu_4N)_4H_3(PW_{11}O_{39})$ was homogeneously dispersed on MCM-41 surface without effecting its mesoporous structure (even at high loadings). Nitrogen adsorption/desorption results showed that the material possessed high surface area (805–912 m^2/g), pore volume (0.65–0.19 cm^3/g) and pore diameter (2.2–3.0 nm). Wide angle XRD and ^{31}P CP–MAS NMR results revealed that $(Bu_4N)_4H_3(PW_{11}O_{39})$ was grafted onto silica surface through W–Si–O linkage and its structural integrity was maintained during preparation method. The material was found to be highly active and reusable catalyst for oxidative desulfurization (ODS) of organosulfur compounds and desulfurized almost 100% of Dibenzothiophene (DBT) and thiophene under mild reaction conditions. When the catalyst was applied for ODS of FCC cycle oil a sulfur removal of 97.2% was achieved.

1. Introduction

The use of environmentally friendly fuels requires the removal of organosulfur compounds present in various petroleum fractions, because these compounds are known to contribute to air pollution, acid rain and also can poison catalytic converters, corrodes parts of internal combustion engines and refinery facilities due to the formation of oxy-acids of sulfur [1,2]. Most of the sulfur compounds in fuels are removed by conventional hydrodesulfurization (HDS) method, but aromatic organosulfur compounds (e.g. Dibenzothiophene (DBT)) are difficult to remove by this technique. However, these hindered compounds can be easily removed by catalytic oxidative desulfurization (ODS) method, which involves oxidation of these organosulfur compounds to sulfoxides and sulfones in the

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