

Support information

One-pot extraction and aerobic oxidative desulfurization with highly dispersed V₂O₅/SBA-15 catalyst in ionic liquids

Chao Wang,^[a] Zhigang Chen,*,^[a] Xiaoyu Yao,^[b] Wei Jiang,^[c] Ming Zhang,^[c]

Hongping Li,^[c] Hui Liu,^[b] Wenshuai Zhu,*,^[b] and Huaming Li^[c]

[a]School of the Environment and Safety Engineering, [b]School of Chemistry and Chemical Engineering, [c]Institute for Energy Research, Jiangsu University, 301 Xuefu Road, Zhenjiang 212013, P. R. China

E-mail: chenzg01@126.com (Zhigang Chen), zhuws@ujs.edu.cn (Wenshuai Zhu)

Experimental Section

Materials

All chemicals in this experiment are of analytical reagent (AR) grade and used without further purification. Dibenzothiophene (DBT), 4-methyldibenzothiophene (4-MDBT) and 4,6-Dimethyldibenzothiophene (4,6-DMDBT) are purchased from Sigma-Aldrich. Decahydronaphthalene, tetradecane, ammonium metavanadate (NH_4VO_3), tetraethyl orthosilicate (TEOS), poly(ethyleneglycol)-*block*-poly(propylene glycol)-*block*-poly(ethylene glycol) (Pluronic P123) and hydrochloric acid (HCl) are purchased from Sinopharm Chemical Reagent Co., Ltd. 1-butyl-3-methylimidazolium tetrafluoroborate ($[\text{Bmim}]\text{BF}_4$, >99%), 1-butyl-3-methylimidazolium hexafluorophosphate ($[\text{Bmim}]\text{PF}_6$, >99%), 1-n-octyl-3-methylimidazolium tetrafluoroborate ($[\text{Omim}]\text{BF}_4$, >99%) and 1-n-octyl-3-methylimidazolium hexafluorophosphate ($[\text{Omim}]\text{PF}_6$, >99%) are purchased from Shanghai Chengjie Chemical Co., Ltd. Actual diesel oil without any desulfurization treatment is purchased from Petro China Co Ltd..

Characterization and analysis

Scanning electron microscopy (SEM) is performed on a FESEM Model JEOL JSM-7001F field with an acceleration voltage of 10 kV. Transmission electron microscopy (TEM) micrographs are taken with a JEOL-JEM-2010 (JEOL, Japan) operated at 200 kV. Then energy dispersed spectrum (EDS) is used to analyze the element of the as-prepared samples. X-ray photoelectron spectroscopy (XPS)

measurements are performed on a PHI 5600 multitechnique system with a monochromatic Al K α X-ray source (Physical Electronics). X-ray powder diffraction (XRD) analysis is carried out on a Bruker D8 diffractometer with high-intensity Cu K α ($k = 1.54 \text{ \AA}$). Raman tests are carried out using Thermo Scientific DXR Smart Raman spectrometer equipped with a 532 nm excitation. The Brunauer–Emmett–Teller (BET) method is utilized to calculate the specific surface areas (S_{BET}). The pore size distribution (PSD) is obtained with using the Barrett–Joyner–Halenda (BJH) model from adsorption data. X-band electron spin resonance (ESR) spectra are recorded at ambient temperature on a JES FA200 spectrometer.

The temperature of the GC process started at 100 °C and increased to 200 °C at 15 °C/min. Injector temperature is 300 °C and detector temperature is 250 °C. The temperature program of the GC-MS process started at 100 °C and rose to 200 °C at 15 °C/min. The injector temperature is 250 °C.

Preparation of model oil

Firstly, model oils with the same sulfur contents (500 ppm) are prepared by dissolving three sulfur compounds, DBT, 4-MDBT, 4,6-DMDBT, in decahydronaphthalene, respectively. Then, model oils with different sulfur content (600, 800 and 1000 ppm) are prepared by dissolving different amounts of DBT in decahydronaphthalene. In addition, tetradecane is used as an internal standard here.

Supplementary Figures

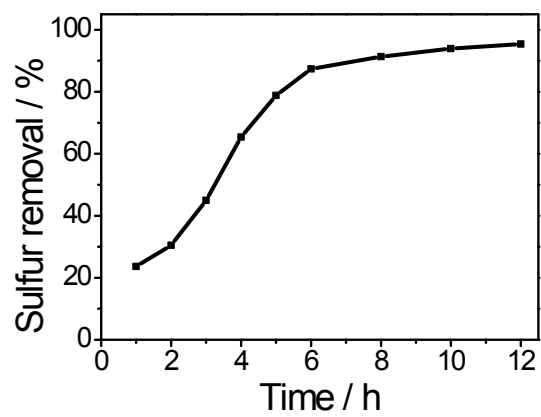


Figure S1 Sulfur removal with actual diesel oil.

Experimental conditions: $T = 120^{\circ}\text{C}$, $V(\text{oil}) = 50 \text{ mL}$, $V([\text{Bmim}]\text{BF}_4) = 10 \text{ mL}$,
 $m(\text{catalyst}) = 0.10 \text{ g}$, $v(\text{air}) = 100 \text{ mL/min}$.

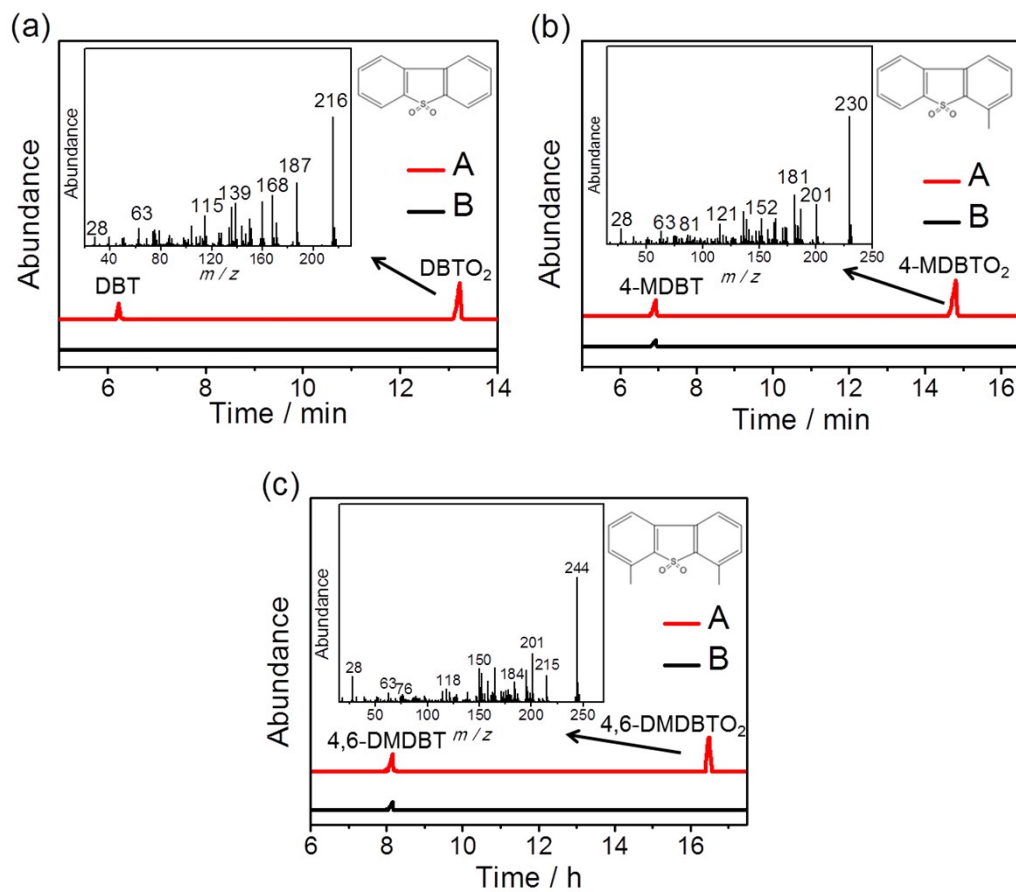


Fig. S2 GC-MS analysis of reacted oil.

A: analysis of IL phase; b) analysis of oil phase.

Table S1 The elements distribution of different elements from XPS analysis

Entry	Elements	Atomic ratio	Mass ratio
1	V	0.93%	2.35%
2	Si	32.14%	44.66%
3	O	66.93%	52.98%

Table S2 Sulfur removal of oils with different ionic liquids

Entry	Type of ionic liquids	Sulfur removal / %
1	[Bmim]BF ₄	99.3
2	[Bmim]PF ₆	64.7
3	[Omim]BF ₄	79.6
4	[Omim]PF ₆	58.4
5	---	23.8

Experiment conditions: T = 120°C, V(oil) = 50 mL, V([Bmim]BF₄) = 5 mL,

m(catalyst) = 0.05 g, t = 4 h, v(air) = 100 mL/min.