Supporting information

for

Solvothermal Synthesis of MoS_2 Nanospheres in DMF-Water Mixed Solvents and Their Catalytic Activity in Hydrocracking of Diphenylmethane

Hui Du,[a] Dong Liu,*[a] Min Li,[a] Raja L. AL Otaibi,*[b] Renqing Lv [c] and Yadong Zhang [a]

a. State Key Laboratory of Heavy Oil Processing, China University of Petroleum, Qingdao 266555, Shandong, P. R. China. E-mail: ldongupc@vip.sina.com; Tel: +86-532-86984629

b. Petrochemical Research Institute, P.O. Box 6086 Riyadh 11442, King Abdulaziz City for Science and Technology, Kingdom of Saudi Arabia. E-mail: raletabi@kacst.edu.sa.

c. College of Science, China University of Petroleum, Qingdao 266555, Shandong, P. R. China.

Material and methods

Ionic liquid-assisted solvothermal synthesis

The ionic liquid [EMIM]Br was synthesized according to the literature.[1] The experiment scheme was arranged as follows. Both 0.5 mmol of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ and 10 mmol of $CS(NH_2)_2$ were dissolved in V_2 ml of distilled water, then 6 ml of [EMIM]Br and V_1 ml of DMF was injected into the above solution under agitation. The total volume of DMF and distilled water (V_1 and V_2) was 54 ml. 12 $mol\cdot L^{-1}$ of hydrochloric acid was used to adjust the pH of solution less than 1. Then the above solution was transferred in a 100 ml Teflon-lined stainless steel autoclave after 30 min of ultrasonic treatment. Solvothermal reaction was carried out at 200 ^oC for 24 h. After chilling, the black precipitates were washed with distilled water and ethyl alcohol three times to remove the unreacted reactants and solvents, and finally dried at 50 ^oC for 4 h in a vacuum oven. The prepared MoS_2 products were annealed in tube furnace at 700 ^oC for 2 h in the mixed atmosphere of N_2/H_2 (9/1, v/v).

Physical characterization

XRD analysis was performed on a PANalyitcal X Pert PRO MPD X-ray diffraction using Cu Kα radiation (λ = 0.15418 nm) with 2θ ranging from 5° to 65°. TEM and HRTEM images were taken with a JEOL JEM-2100F transmission electron microscope operated at an accelerating voltage of 200 KV. A Japan Hitachi S-4800 field-emission scanning electron microscope were used to obtain the SEM images of as-prepared MoS_2 samples. The Brunauer-Emmett-Teller (BET) surface area measurements were carried out by N_2 adsorption at 77 K using a Quantachrome NOVA 3000 system.

Catalytic activity

Diphenylmethane (DPM, 99%), which was obtained from Aladdin Industrial Inc. and used without further purification, was used to appraise the catalytic activity of the prepared MoS_2 samples. In each experiment, 30 g of DPM and 0.5 g of MoS_2 samples were loaded in a 100 ml batch-type autoclave with an electromagnetic stirrer. After being purged by hydrogen for 3 times, the autoclave was charged with hydrogen to 5.0 MPa at room temperature. Subsequently, the autoclave was heated to reaction temperature in 30 min and kept for 1 h. At the end of each experiment, the autoclave was cooled rapidly to room temperature in water, and the residual hydrogen was vented out. The composition of hydrocracking products was quantified by a Varian CP3800 gas chromatography (GC). DPM hydrocracking with different catalysts were performed 3 times, and the values reported were the average values.

References

1 K. Su, X. Liu, M. Ding, Q. Yuan, Z. Li and B. Cheng, *J. Mol. Catal. A-Chem.*, 2013, **379**, 350-354.