

## TEM Characterization of Co-Mo-W HDS Catalysts Supported on Siliceous SBA-16 Material

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The hydrotreatment of middle distillates is an important process in the petrochemical industry [1]. Typically, the hydrotreatment reactions are catalyzed by Co(Ni)Mo(W) catalysts supported on alumina [2].

The origin of the almost exclusive use of alumina as support has been ascribed to its outstanding textural and mechanical properties and its relatively low cost [3]. However, the evidence of undesirable strong metal-support interaction has urged numerous studies devoted to new supports [3]. Recently, the use of mesoporous silica materials as SBA-16 for supporting CoMo, NiMo and trimetallic NiMoW as active phase in hydrodesulphurization (HDS) processes are intensively studied [4]. The advantages of mesoporous materials include high surface-to-volume ratio, variable framework compositions and high thermal stability.

The siliceous SBA-16 mesoporous material was synthesized according the procedure described by Flodström and Alfredsson [5]. Pluronic F127 triblock copolymer was used as the structure directing and the corresponding amount of water and HCl 2M were mixed under stirring. After dissolution, TEOS was added and allowed to react at room temperature for about 24 h. Then, the mixture was transferred into polypropylene bottles and heated at 80 °C for 48 h. The solid residue was filtered, washed and dried at room temperature and then at 110 °C was dry for 24 h. Finally, the sample was calcined at 550 °C in air for 6 h. Each support was loaded with fixed amounts of molybdenum (8.53 wt% as MoO<sub>3</sub>), tungsten (13.75 wt% as WO<sub>3</sub>) and cobalt (3.81 wt% as CoO), by simultaneous impregnation via incipient wetness method. For this, an aqueous solution containing ammonium heptamolybdate tetrahydrate ((NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O, Aldrich), ammonium metatungstate ((NH<sub>4</sub>)<sub>6</sub>H<sub>2</sub>W<sub>12</sub>O<sub>40</sub>·xH<sub>2</sub>O, Aldrich) and cobalt nitrate hexahydrate (Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O, Aldrich, 98%) was prepared.

Since metal sulfides are the active phases in HDS reaction, the fresh sulfided catalysts were studied by TEM. It is known that both MoS<sub>2</sub> and WS<sub>2</sub> phases adopt a layered structure with a Mo(W) atoms situated in trigonal prismatic coordination sphere. As expected, after sulfidation all catalysts display the typical fringes of MoS<sub>2</sub> (WS<sub>2</sub>) crystallites. The location of MoS<sub>2</sub> (WS<sub>2</sub>) fringes on the SBA-16 substrate is shown in Fig. 1.

For all catalysts, the arrangement of W(Mo)S<sub>2</sub> layers parallel to the substrate indicates the basal plane attachment. No edge-plane attachment was observed because such species easily escape detection. In general, the SBA-16-based system show high W(Mo)S<sub>2</sub> slabs density with disordered curved fringes of the W(Mo)S<sub>2</sub> such as show in the Fig. 1.

## References

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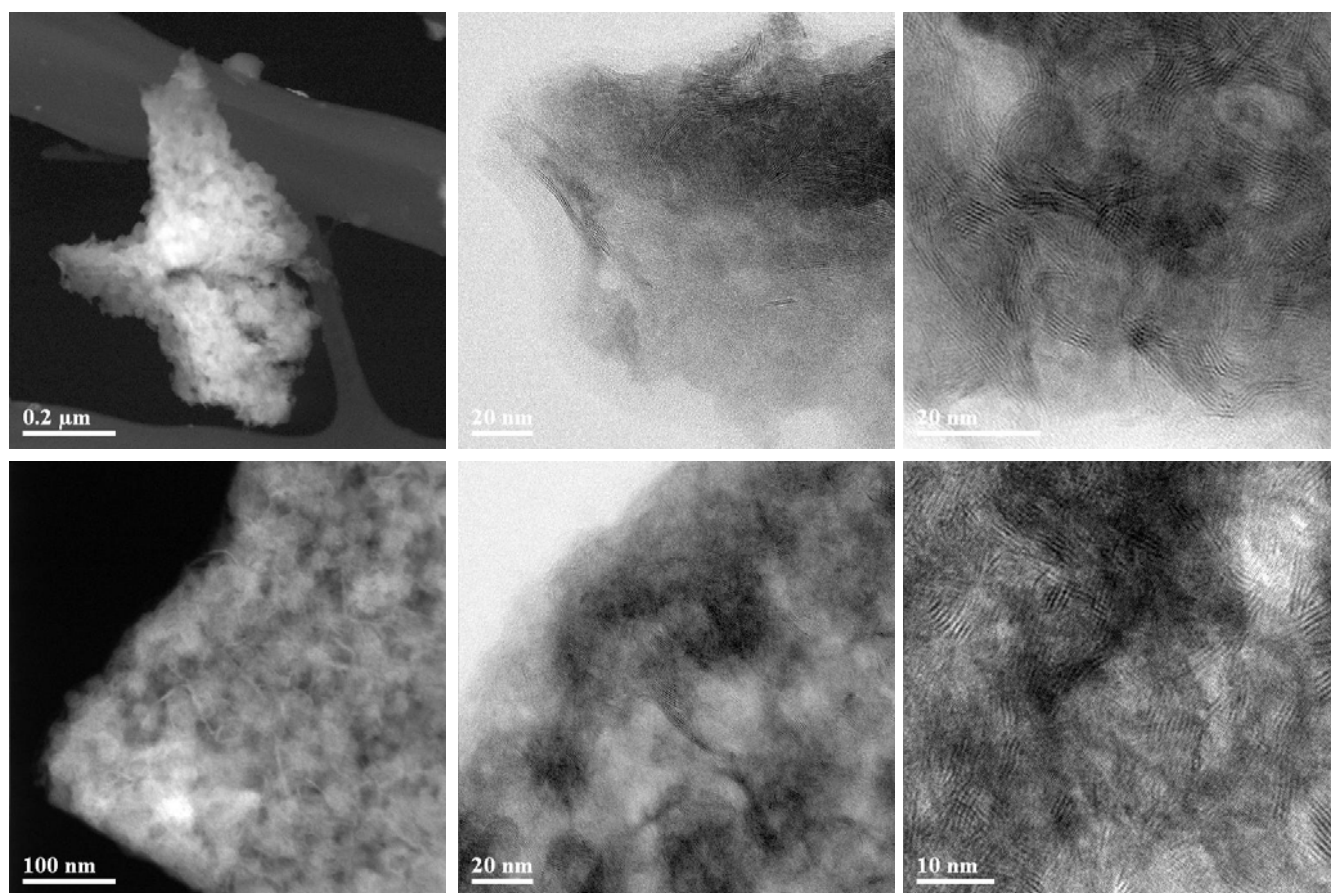


FIG. 1. TEM images of CoMoW supported on SBA-16 substrate



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