ORIGINAL PAPER



# Influence of HZSM-5 content on behavior of CoMo/HZSM5-HMS composite catalysts in hydrodesulphurization of dibenzothiophene

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**Abstract** CoMo-supported micro/mesoporous catalysts (CoMo/HZSM5(x)-HMS) were synthesized by varying the weight percent of zeolite (10, 20, 30 and 40 wt%). Catalytic functionalities were investigated for hydrodesulphurization reaction of dibenzothiophene over a wide temperature range of 250–400 °C. Characterization methods as XRD, XRF, FT-IR, UV–Vis DRS, NH<sub>3</sub>-TPD, H<sub>2</sub>-chemisorption, nitrogen adsorption–desorption and TGA confirmed the obtained results from these catalysts. A significant enhancement of the catalytic activity was observed for the catalysts supported on CoMo/HZSM5(20)-HMS at T = 350 °C (>97%).

**Keywords** Micro/mesoporous catalysts · Hydrodesulphurization · Dibenzothiophene · Catalytic

activity

# Introduction

Due to strict environmental legislations to reduce sulfur from natural gas and refined petroleum products, hydrodesulphurization (HDS) is widely used as a catalytic chemical process to remove sulfur [1–3]. For this reason, the production of more active HDS catalysts is one of the main targets in the refining industry [4, 5]. To generate an active catalyst

N. Parsafard n\_dastmoozeh@sbu.ac.ir for this process, it needs to know its properties and relationship to the nature of the support, composition, preparation method, promoters, additives and other variables. It has been demonstrated that a support can affect the dispersion of active phase, interactions between metal-support and metal-promoter and thus can alter the catalytic properties. For this process, molybdenum or tungsten is usually used as an active phase and cobalt or nickel is added as a promoter [6]. Since supports have a large influence on the catalytic activity, many studies have been done on various supports to achieve the best result in the removal of sulfur using the HDS reaction [7–9].

During the past decades, mesoporous materials with ordered pore structures, large pore diameters and high surface areas like FSM-16 [6], HMS [10–12], KIT-6 [3, 4, 15], SBA-15 [13–17], MCM-41 [18] and so on, and their substituted analogs attracted widespread attention as potential support materials [19, 20]. These materials have controllable pore size, but they involve poor hydrothermal stability and low reactivity as compared to employed zeolites for this reaction [12].

So in recent years, there has been great interest in composite supports due to their high ability in the catalytic applications. These micro/mesoporous composite materials have developed significant advances in the improvement of acidity and stability [21]. Many micro/mesoporous materials, such as MSU/S [22], Beta/MCM-41 [23] and Beta/ SBA-15 [24] were synthesized and used for various catalytic reactions [25, 26]. According to our knowledge, there are no focused reports on combining the particular advantages of HZSM-5 and HMS in this process and especially investigating the effects of HZSM-5 contents on the characteristics and the catalytic activities of these composite HDS catalysts. Hence, in the present research, CoMo catalysts were prepared using the micro/mesoporous supports

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(HZSM-5/HMS) with different amounts of zeolite and were applied in desulfurization reaction of dibenzothiophene.

# Experimental

## Synthesis and preparation of materials

A series of micro/mesoporous composite supports (HZSM5/HMS) with various amounts of HZSM-5 were prepared by a method similar to our published procedure [26]. The molybdenum- and cobalt-promoted catalysts were prepared by successive impregnation method using these supports and appropriate amounts (3 wt% Co and 10 wt% Mo) of ammonium heptamolybdate tetrahydrate (as a molybdenum source) and cobalt nitrate hexahydrate (as a cobalt source). The impregnated catalysts were dried in an oven at 110 °C overnight followed by calcination at 300 °C for 4 h. Prior each reactor test, the calcined catalysts were pretreated and sulfurized at 400 °C for 2 h in a CS<sub>2</sub>/H<sub>2</sub> flow (40 mL min<sup>-1</sup>).

Hereafter, the above micro/mesoporous samples would be denoted as CoMo/ZH-x that x is HZSM-5 weight percent and is equal to 10, 20, 30 and 40 versus wt%.

### Characterization of the samples

For measuring the metal contents, the synthesized samples were characterized by XRF (XRF-8410 Rh 60 kV).

X-ray diffraction patterns (XRD) were recorded by X-PERT diffractometer Cu–K $_{\alpha}$  radiation (45 kV and 50 mA).

A BOMEM FT-IR spectrophotometer model Arid-Zone TM, MB Series, was used to obtain the FT-IR spectra, surface characterization for the dehydroxylation and removal of the residual surfactants from the dried samples. This analysis was performed in the wave numbers range from 4000 to 400 cm<sup>-1</sup>.

The UV–Visible diffuse reflectance spectra were recorded by a Shimadzu UV-2100 spectrophotometer equipped with a diffuse reflectance attachment. The spectra were collected in the 200–800 nm range at room temperature with  $BaSO_4$  as a reference.

The specific surface area, pore volume and pore size distributions were measured by  $N_2$  physisorption (BET) at -196 °C by High-Speed Gas Sorption Analyzer (NOVA2200) version 7.11 using a standard procedure.

To evaluate the acidity, these samples were analyzed by a TPD/TPR analyzer (2900 micromeritics) equipped with a TCD sensor. The obtained profiles of  $NH_3$  desorption were recorded from 25 to 800 °C under a heating rate of 10 °C min<sup>-1</sup>. Also, hydrogen pulse chemisorption was performed to measure the metal dispersion on the supports. Scanning electron microscopy (SEM) analyzes were conducted on a HITACHI S-4160 instrument operating at an accelerating voltage of 30 kV over the coated samples with gold prior to SEM measurements.

The amounts of coke laid down on the surfaces of the spent composites were measured by thermogravimetric analysis TGA/STA503 M instrument. In this analysis, the weight changes in the coked samples during oxidation were measured. The coke was calcined by heating rate of 10 °C min<sup>-1</sup> to a final temperature of 800 °C in flowing air.

### Catalytic performance evaluation

Hydrodesulphurization of dibenzothiophene was carried out at a temperature range of 250–400 °C and 1 atm pressure in a continuous fixed-bed microreactor packed with 1.0 g of each sulfurized composite. The liquid reactant (dibenzothiophene/*n*-heptane mixture with 5% by volume dibenzothiophene) was fed into the reactor with a space velocity (LHSV) 1.0 h<sup>-1</sup>. Hydrogen was also introduced in a best H<sub>2</sub>/HC molar ratio for our samples to get the best selectivity of desired products (H<sub>2</sub>/HC = 7). The reaction products were analyzed by online gas chromatography (Agilent Technologies 7890A) equipped with an FID detector.

# **Results and discussions**

### X-ray diffraction (XRD) patterns

To reveal whether or not the structures of HMS and HZSM-5 were preserved after impregnation of metal



Fig. 1 XRD patterns for calcined CoMo-supported samples. (calcination: 300  $^\circ C$  for 4 h)

Table 1Physicochemicalproperties of CoMo-supportedsamples. (calcination: 300 °C

for 4 h)

oxide precursors, the XRD analysis was performed (Fig. 1). In all XRD patterns, it can be seen the peaks for HMS structure ( $2\theta = 2.3^{\circ}$  [27]) and HZSM-5 phases  $(6-11^{\circ} \text{ and } 22-25^{\circ} \text{ [28]})$ . The amorphous part of the substrate also is observed between 20° and 30°. Compared with pure supports, the XRD patterns reveal that the diffraction peaks become less intense, broader and shift to higher angles. These data indicate a partial lattice collapse upon metals (Co and Mo) incorporation [29]. Table 1 shows the *d*-spacing  $(d_{100})$ , unit cell parameter  $(a_0)$  and wall thickness (wt) values that have been calculated from XRD data. These values decrease with increasing HZSM-5 content and after incorporating Co and Mo species. It should be noted the metal or metal alloy in these XRD patterns does not show any peak that it shows their good dispersion.

# Fourier transform infrared (FT-IR) spectroscopy results

The FT-IR spectra of the CoMo samples were shown in the  $4000-400 \text{ cm}^{-1}$  range (Fig. 2). These spectra were compared with the pure supports spectra which were given in our previous work [26] and so not shown here. In all



Fig. 2 FT-IR spectra of the framework vibration for the calcined powder samples. (calcination:  $300 \text{ }^{\circ}\text{C}$  for 4 h)

spectra, the characterization bands for HMS and HZSM-5 as the pure supports were observed [26].

The region of ~1085–500 cm<sup>-1</sup> is important for characterization of molybdates. The bonds in ~1050–900 and ~850–700 cm<sup>-1</sup> ranges represent terminal Mo = O and asymmetric Mo–O–Mo and O–Mo–O stretching

Samples	HMS	HZSM5	ZH-10	ZH-20	ZH-30	ZH-40
Surface properties						
$S_{\rm BET} ({ m m}^2 { m g}^{-1})$	714	256	712	711	619	524
$V_{\rm p}^{\rm a}({\rm cm}^3{\rm g}^{-1})$	0.72	0.17	0.84	0.79	0.71	0.67
$S_{\rm micro} ({\rm m}^2 {\rm g}^{-1})$	-	188	24	48	74	94
$V_{\rm micro} ({\rm cm}^3{\rm g}^{-1})$	-	0.10	0.09	0.15	0.20	0.25
$d_p$ (nm)	4.03	2.66	4.72	4.44	4.59	5.11
NS <sup>b</sup> <sub>BET</sub>	0.90	0.65	0.90	0.90	0.87	0.83
$M_{ m d}^{ m c}$	17.8	16.3	15.5	22.1	20.9	14.8
Si/Al <sup>d</sup>	$\infty$	13.03	65.51	60.09	54.13	49.11
Strength of acid sites (mmol N	VH <sub>3</sub> /g <sub>cat</sub> )					
Weak acid (100-300 °C)	1.15	1.30	1.17	1.23	1.27	1.32
Strong acid (300-600 °C)	-	0.95	0.60	0.63	0.79	0.83
Total acid	1.15	2.25	1.77	1.86	2.06	2.15
Lattice properties						
$d_{100}^{\rm e}({\rm nm})$	4.51	3.24	4.26	4.23	4.16	3.51
$a_0^{\rm e}$ (nm)	5.21	3.74	4.92	4.89	4.80	4.05
wt <sup>e</sup> (nm)	1.95	~2.24	2.58	2.57	2.51	1.77

 $a_0$  calculated by equation  $a_0 = d_{100} \times 2/\sqrt{3}$  and we was evaluated by subtracting pore diameter  $(d_p)$  from the unit cell parameter [29]

<sup>a</sup> Using BJH method

<sup>b</sup> NS<sub>BET</sub> is normalized  $S_{BET}$  and calculated from equation [29, 30]: NS<sub>BET</sub> =  $S_{BET}$  of catalyst/ [(1-y) ×  $S_{BET}$  of support]; y is the weight fraction of the guest phases

<sup>c</sup> Metal dispersion using H<sub>2</sub>-chemisorption method

<sup>d</sup> Using XRF method

<sup>e</sup>  $d_{100}$  as calculated from the XRD patterns

vibrations, respectively. The tetrahedral, octahedral and other poly molybdates and crystalline  $MoO_3$  create some features around ~930, 980 and 580 cm<sup>-1</sup>. Also the octahedral and tetrahedral molybdenum species make the bands between ~1085 and ~800 cm<sup>-1</sup>. The vibrations of Mo tetrahedral, octahedral and other polyhedral species appear at ~930–830, 990–930 and 860–800 cm<sup>-1</sup>, respectively. These characteristic bands normally overlap with the characteristic bands of HMS and HZSM-5. Therefore, it is difficult to ascertain the bands [4].

## UV-Vis diffuse reflectance spectroscopy (UV-Vis DRS)

UV-Vis DRS was used to find the symmetry of the CoMo species and information about the distribution of oxide species or their dispersion over the supports (Fig. 3). The CoMo/HMS spectrum shows the bands in the ranges of 200-400 and 450-700 nm. These bands in the spectrum of CoMo/HZSM-5 have low intensity. The bands in the 220-270 and 300-330 nm ranges, which are observed in all spectra, are assigned to  $O^{2-}-Mo^{6+}$  ligand to metal charge transfer transition in isolated tetrahedral coordination  $MoO_4^{2-}$  and octahedral  $Mo^{6+}$  species, respectively [29]. A second peak at around 230 nm is related to both tetrahedral and octahedral Mo<sup>6+</sup> species. The mixtures of both tetrahedral and octahedral Mo<sup>6+</sup> species are present in all spectra. It seems that the contribution of octahedral Mo<sup>6+</sup> species increases in intensity and becomes almost distinguishable shoulder of the tetrahedral Mo<sup>6+</sup> species absorption [29]. It can be concluded that tetrahedral  $Mo^{6+}$ species are better dispersed over these samples. Besides, the absorption spectra of CoMo/HMS and CoMo/ZH composite catalysts in UV region are more symmetrical than the CoMo/HZSM-5 catalyst. It shows the dispersion of Mo in the presence of HMS phase is much higher than the ZSM-5 pure phase. All samples present a band in the ~480-720 nm range of the visible region, which are related to Co species. This broad peak is characterized as the tetrahedrally and octahedrally coordinated  $Co^{2+}$  [12].  $CoMoO_4$  species are as an octahedral component and the Co<sup>2+</sup> species interacting with the support are as a tetrahedral component. According to the literature [29], the UV-Vis DRS bands at about 400-450 and 700-750 nm, could be ascribed as the  $Co_3O_4$  phase with octahedral Co (III). These bands were not observed in any of spectra that it shows a higher dispersion of the Co species. The effect of cobalt promoter addition was reported as a blue shift of the Mo absorption and an increase in the dispersion of octahedral Mo species or the change in the coordination of the Mo oxide species from octahedral to tetrahedral [12, 31, 32].



Fig. 3 UV–Vis DRS of CoMo samples supported on HMS, HZSM-5 and ZH-x. (calcination: 300 °C for 4 h)



Fig. 4 Nitrogen adsorption–desorption isotherms for CoMo-supported samples. (calcination:  $300 \text{ }^\circ\text{C}$  for 4 h)

#### **Textural properties**

The nitrogen physisorption isotherms of the samples are showed in Fig. 4 and listed in Table 1. N<sub>2</sub> adsorption–desorption isotherms of the CoMo/supports indicate surface areas that changed between 524 and 712 m<sup>2</sup> g<sup>-1</sup> indicating that the incorporations of Co and Mo display a significant effect into the frameworks of composites (Table 1). When molybdenum co-impregnates with cobalt, the lower surface areas were obtained as compared with the surface areas of HZSM5(x)/HMS supports [26]. According to the results, it seems that the incorporations of Co and Mo metals block the micropores of the supports and so surface areas and pore volumes decrease. These incorporations are also due

to partial loss of structural ordering of the channels. In Table 1, the  $NS_{BET}$  values were also calculated for the prepared samples. These amounts suggest a very good dispersion of the supported species.



Fig. 5  $NH_3$ -TPD profiles of the calcined CoMo samples supported on the ZH-10, ZH-20, ZH-30 and ZH-40 materials. (calcination: 300 °C for 4 h)

### NH<sub>3</sub>-TPD results

NH<sub>3</sub>-TPD method was used to measure the acidity of samples. The effects of zeolite content and metal oxides on the acidity of samples were exhibited in Fig. 5 and listed in Table 1. As expected, the acidity increased with increasing HZSM-5 content. On the other hand, the overall acidity of modified composites, in compared with pure supports [26], increased with an addition of cobalt and molybdenum metals. According to the literature [33], it has been reported that due to the complex formation by  $Co^{2+}$  ions and the generation of acid sites with the cobalt–molybdenum oxide, the acidity increases.

### Scanning electron microscopy (SEM)

Figure 6 shows SEMs of the CoMo composites. As observed, the primary structures that were seen in the pure composites [26], undergo decomposition during the incorporation of Co and Mo metals.

### Catalytic activity in the HDS of dibenzothiophene

Table 2 shows the data of conversion of dibenzothiophene (DBT) over CoMo/HMS, CoMo/HZSM-5, CoMo/ZH-10, CoMo/ZH-20, CoMo/ZH-30 and CoMo/ZH-40 samples



Fig. 6 Scanning electron micrographs of CoMo/ZH samples. (calcination: 300 °C for 4 h; *scale bars* are 5, 10 and 20  $\mu$ m for (**a**), (**b**) and (**c**) images, respectively)

**Table 2** Conversion and BP and CHB yields for various samples at different temperatures. (calcination: 300 °C for 4 h; time on stream in reactor test: 1 h)

Table 3 Conversion, BP and CHB yields and coke amounts at 350  $^{\circ}\mathrm{C}$  for various times

Samples	Temperature (°C)	Conversion (%)	Yield (%)	
			BP	CHB
CoMo/HMS	250	56.3	54.2	2.1
	300	61.2	59.0	2.2
	350	75.8	72.5	3.3
	400	71.2	67.3	3.9
CoMo/HZSM-5	250	54.1	52.1	2.0
	300	60.1	57.9	2.2
	350	71.4	68.3	3.1
	400	69.8	66.0	3.8
CoMo/ZH-10	250	45.8	44.1	1.7
	300	52.7	50.8	1.9
	350	66.3	63.4	2.9
	400	59.9	56.6	3.3
CoMo/ZH-20	250	63.4	61.0	2.4
	300	74.3	71.6	2.7
	350	97.1	92.9	4.2
	400	80.1	75.7	4.4
CoMo/ZH-30	250	56.3	54.2	2.1
	300	62.9	60.6	2.3
	350	92.6	88.6	4.0
	400	78.2	73.9	4.3
CoMo/ZH-40	250	42.9	41.3	1.6
	300	45.6	44.0	1.6
	350	65.9	63.0	2.9
	400	58.9	55.7	3.2

Samples	Time (h)	Conversion (%)	Yield (%)		Coke amounts
			BP	CHB	(wt%)
CoMo/HMS	2	71.1	68.5	2.6	0.9
	4	70.7	68.2	2.5	
	6	70.1	67.1	3.0	
	8	69.5	65.7	3.8	
CoMo/ HZSM-5	2	63.1	60.8	2.3	12.5
	4	50.9	49.1	1.8	
	6	45.7	43.7	2.0	
	8	44.6	42.2	2.4	
CoMo/ ZH-10	2	63.8	61.4	2.4	2.1
	4	60.2	58.0	2.2	
	6	59.4	56.8	2.6	
	8	57.8	54.6	3.2	
CoMo/ ZH-20	2	90.6	87.2	3.4	3.2
	4	84.3	81.3	3.0	
	6	81.5	78.0	3.5	
	8	79.8	75.4	4.4	
CoMo/ ZH-30	2	88.3	85.0	3.3	3.4
	4	80.3	77.4	2.9	
	6	76.5	73.2	3.3	
	8	74.9	70.8	4.1	
CoMo/ ZH-40	2	60.5	58.3	2.2	5.3
	4	53.3	51.4	1.9	
	6	51.2	49.0	2.2	
	8	50.3	47.6	2.7	

for 1 h on stream at each temperature in the range of 250–400 °C. Under our experimental conditions, the only detectable products for hydrodesulphurization of dibenzothiophene were biphenyl (BP) and very low amounts of cyclohexylbenzene (CHB). Obtaining BP as a main product shows the direct hydrogenolysis or direct desulfurization (DDS) pathway [33]. CHB product is achieved from a hydrogenation of the rings prior the desulfurization (HYD) pathway through to tetra-hydro-dibenzothiophene (THDBT) intermediate [33].

As seen in Table 2, the CoMo samples at 350 °C show the highest catalytic activity and then this high catalytic activity decreases. This decline in activity is probably due to the loss of sulfur by the over-reduction [2] and the increase in severe side reactions such as cracking and reforming up to 350 °C. The most active of the prepared composite for the dibenzothiophene hydrodesulphurization is CoMo/ZH-20 at 350 °C; whereas, the lowest conversion is observed for CoMo/ZH-40 at 250 °C. The results show that the activity of the samples does not follow their acidic properties (Table 1). This may be related to the sintering effect of  $MoS_2$  phase on the catalytic activity and its subsequent migration from the surface to the bulk [1].

In order to study the stability of the composites as a function of the time on stream, the catalytic HDS test over these materials was performed at 350 °C during 8 h. In accordance with the results summarized in Table 3, significant deactivation and coke formation (by TG/DTA) were observed for CoMo/HZSM-5 and then CoMo/ZH-40. As deduced from these results, the coking amount increases with increasing the catalytic acidity.

### Conclusion

CoMo/ZH composites were prepared by co-impregnation method. The characterization results showed that ZH composites were successfully synthesized. The textural and acidity properties of these composites improved HDS reaction compared with those of HZSM-5 and HMS. The influence of temperature and time on stream was investigated in the HDS of dibenzothiophene. The catalytic performance results showed that CoMo/ZH-20 has the highest HDS activity among all the prepared composites (>97% at 350  $^{\circ}$ C). Results presented that the metals dispersion is a more effective factor than acidity in HDS reaction.

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