Electronic Supplementary Information

Role of transition metals on MoS_2-based supported catalysts for hydrodeoxygenation (HDO) of Propylguaiacol

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Kraft lignin liquid product analysis (2D GC × GC-MS-FID analysis)

The individual product selectivity in the liquid was calculated by dividing the corresponding MS blob volume of the product by the total MS blob volume for all identifiable products in the liquids.

The bio-liquid products were also analyzed by GC × GC-MS-FID on Agilent 7890B gas chromatograph equipped with an oven, a flow splitter, a modulator, and a flame ionization detector. The injector temperature was 280 ˚C and the sample injection volume was 1 µL. Helium gas was used as a carrier gas with a flow rate of 1 mL/min with a split ratio of 30. The chromatographic separation involves two columns: a mid-polar phase column VF-1701MS (30 m × 250 µm × 0.25 µm) and a non-polar phase column DB-5MS UI (1.2 m × 150 µm × 0.15 µm). Modulation time on the modulator is 8 s. The oven temperature was initially set at 40 ˚C for 1 min and then heated up to 280 ˚C at a rate of 2 ˚C/min. The flame ionization detector temperature was set at 250 ˚C. The hydrogen flow rate is 30 mL/min and the airflow rate is 350 mL/min. The analysis was performed using the GCImage software for multidimensional chromatography.

Table S1: State of the art sulfided catalysts for HDO of phenolics.

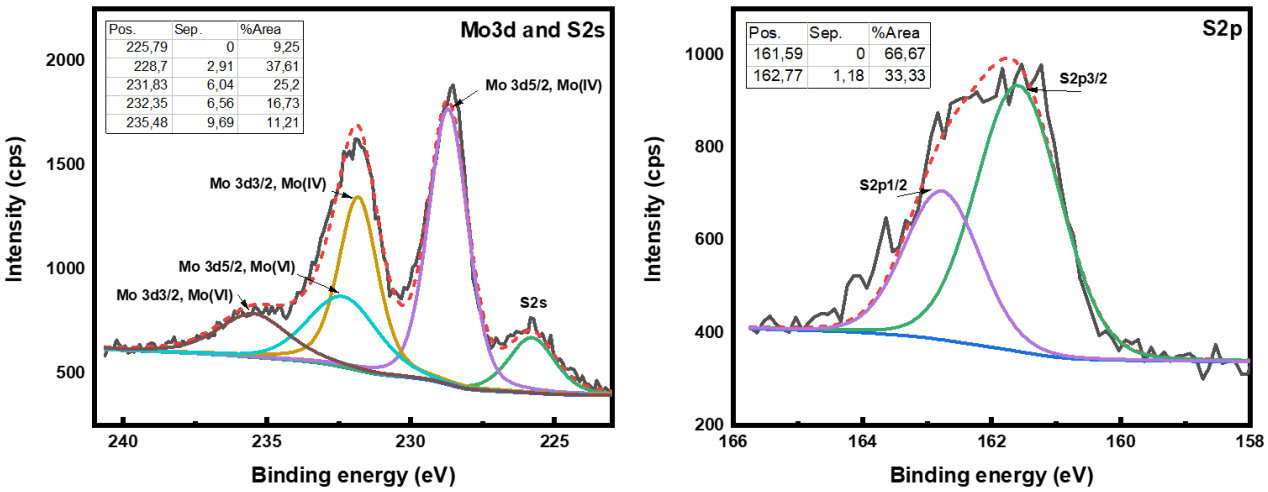
Entry	Sulfided catalysts	Model compound (Phenolics)	Solvent	Conversion (%)	Reaction parameters (reactor type, reaction time, temperature, and pressure)	Major products (selectivity %)	Ref
1	MoS_2	4-propylguaiacol	Dodecane	99.2	Batch, 6 h, 300 °C and 40 bar	Propylcyclohexane (5%), 4-Propylcyclohexene (2.9%), Propylbenzene (57.4%) and 4-Propylphenol (34.7%)	1
2	NiS_2-MoS_2	4-propylguaiacol	Dodecane	99.7	Batch, 6 h, 275 °C and 40 bar	Propylcyclohexane (31.6%), 4-Propylcyclohexene (0.6%), Propylbenzene (47.4%) and 4-Propylphenol (20.4%)	1
3	CoS_2-MoS_2	4-propylguaiacol	Dodecane	100	Batch, 6 h, 250 °C and 40 bar	Propylcyclohexane (13.4%), 4-Propylcyclohexene (0.6%), Propylbenzene (83.7%) and 4-Propylphenol (2.3%)	1
4	Commercial sulphided NiMo/γAl_2O_3	Phenol	Dodecane	-	Continuous, WHSV = 29/ 36 h^{-1}, 200/ 220/ 250 °C and 30 bar	Cyclohexene (traces), Cyclohexane (93.4%) and benzene (6.5%) for 200 °C and 29 h^{-1}	2
5	Commercial reduced NiMo/γAl_2O_3	Phenol	Dodecane	100	Continuous, WHSV = 11/ 21 h^{-1}, 130/ 145 °C and 30 bar	Cyclohexene (<2.5%), Cyclohexane (10%) and Cyclohexanol (90%) for 130 °C and 11 h^{-1}	2
6	CoMoS	p-cresol	Decalin	100 after 3 h	Batch, 4 h, 300 °C and 40 bar	Arene yield (98%)	3
7	CoMo/Al_2O_3-TiO_2	Phenol	Dodecane	93	Batch, 4 h, 320 °C and 54 bar	Benzene (65%), Cyclohexane (25%) and Cyclohexene (3%)	4
8	RuS_2/SBA-15	Phenol	Decalin	9	Continuous, WHSV = 1.28 h^{-1}, 310 °C, TOS = 4 h and 30 bar	Cyclohexane (40%), Cyclohexene (54%), Benzene (3%) and Cyclohexanol (3%)	5
9	Ir-RuS_2/SBA-15	Phenol	Decalin	41	Continuous, WHSV = 1.28 h^{-1}, 310 °C, TOS = 4 h and 30 bar	Cyclohexane (63%), Cyclohexene (11%), Benzene (7%) and Cyclohexanol (19%)	5
10	Pt-RuS_2/SBA-15	Phenol	Decalin	37	Continuous, WHSV = 1.28 h^{-1}, 310 °C, TOS = 4 h and 30 bar	Cyclohexane (62%), Cyclohexene (11%), Benzene (11%) and Cyclohexanol (16%)	5
11	Ni/W/TiO_2	Guaiacol	n-decane	100	Batch, 2.5 h, 300 °C and 70 bar	Phenol (37%), Cyclohexane (16%), Benzene (1%), Creasol (3%) and others (43%)	6
12	NiMo/Al_2O_3	2-ethylphenol	toluene	23	Continuous, 1.11 min (contact time), 340 °C and 70 bar	Oxygenated compounds (19.1%) and deoxygenated compounds (80.9%)	7
13	CoMo/Al_2O_3	2-ethylphenol	toluene	24	Continuous 1.15 min (contact time), 340 °C and 70 bar	Oxygenated compounds (27.7%) and deoxygenated compounds (72.3%)	7

14	Mo/Al_2O_3	2-ethylphenol	toluene	22	Continuous 2.3 min (contact time), 340 °C and 70 bar	Oxygenated compounds (48.4%) and deoxygenated compounds (51.6%)	7
15	NiWS supported on TiO_2, ZrO_2, and TiO_2-ZrO_2	Guaiacol	Hexadecane	100	Batch, 320 °C, 55 bar H_2, and 1000 rpm	80% cycloalkanes (NiWS-TiO_2)	8
16	CoMoS supported on Al_2O_3	Phenol/2-ethylphenol	Toluene	-	Continuous, 400 °C, 70 bar	HDO activity (29.1 mmol. $h^{-1}g^{-1}$) for phenol and (22 mmol.$h^{-1}g^{-1}$) for 2-ethylphenol	9
17	NiMoS/CoMoS supported on γ-Al_2O_3	Guaiacol/Phenol	m-xylene /n-hexadecane	30-100	Continuous/batch, 200-350 °C, 75-80 bar	Cycloalkanes (55% for NiMo and 45% for CoMo) at 300 °C	10
18	CoMoS supported on Al_2O_3	Phenol, o-cresol, anisole, 4-methylanisole, catechol, guaiacol, 4-methylguaiacol,1,3-dimethoxybenzene, syringol, and vanillin.	Dodecane	25-90	Batch, 4 h, 300 °C and, 50 bar	See ref[11]	11
19	NiMoS supported on γ- Al_2O_3	Phenol and methyl heptanoate	Dodecane	100	Batch, 200/250 °C, and 75 bar	Cyclohexane (85%), cyclohexyl cyclohexane (14%), and others (1%)	12
20	CoMoP/MgO	Phenol	Supercritical hexane	17-90	Batch, 350-450 °C, 1 h and 50 bar	Benzene (65%) and other (26%) at 450 °C	13
21	NiMoS/CoMoS supported on γ-Al_2O_3	Phenol and methyl heptanoate	m-xylene	5-28	Batch/continuous, 250 °C, 1 h and 15 bar	See ref[14]	14
22	CoMoS supported on Al_2O_3	Methyl-substituted phenols	n-heptane/n-decane	10-50	Continuous, 300 °C and 28.5 bar	See ref[15]	15
23	CoMoS on activated carbon	Guaiacol, ethyldecanoate, and 4-methylacetophenone	-	17-19	Continuous, 270 °C and 75 bar	See ref[16]	16
24	MoS_2 on activated carbon	Guaiacol	Decalin	10-30	Batch, 300 °C, 50 bar, and 1000 rpm	See ref[17]	17
25	MoS_2 on activated carbon	Guaiacol	Dodecane	55	Batch, 300 °C, 50 bar, 5 h and 1000 rpm	Phenol (52%), Cycloalkanes (12.2%), cyclohexanol (5%), anisole (0.3%), benzene (0.4%), catechol (1.8%), veratrole (0.8%), methanol (0.04%) and gases.	18
26	ReS_2/SiO_2	Guaiacol and phenol	Hexadecane and dodecane	15-20	Batch, 250 °C, 50 bar, and 4 h	For guaiacol, phenol (13%), catechol (1%), and cyclohexanol (0.5%)	19
27	ReS_2/SiO_2	Guaiacol	Dodecane	80	Batch, 300 °C, 50 bar, and 4 h	For ReS_2/SiO_2, phenol (60%), cyclohexane (20%) and others.	20
28	ReS_2/activated carbon	Guaiacol	Dodecane	40-80	Batch, 300 °C, 50 bar, and 4 h	See ref[21]	21
29	$ReS_2/SiO_2(Al_2O_3)$	Guaiacol and 4,6-dimethyldibenzothiophene	Dodecane	80	Batch, 300 °C, 50 bar, and 4 h	See ref[22]	22
30	Re/ZrO_2 and Re/ZrO_2-sulphated	Guaiacol	Decaline	10-70	Batch, 300 °C, 50 bar, and 4 h	See ref[23]	23
31	CoS_2/MoS_2	Creosol and phenol derivatives	Dodecane	18-98	Batch, 250 °C, 40 bar, and 1 h	For CoMo-0.3, toluene (99%)	24
32	MoS_2 and $CoMoS_2$	Phenol	n-decane	30-98	Batch, 350 °C, 28 bar, 150 rpm, and 1 h	See ref[25]	25

33	Amorphous NiMoS	Phenol	n-decane	34.5-96.2	Batch, 350 °C, 28 bar, 150 rpm, and 1 h	For NiMoS-0.3, benzene (30.4%), cyclohexane (52.4%), cyclohexene (9.8%), cyclohexanone (7.4%)	26
34	MoS_2	Phenol, 4-methylphenol, and 4-methoxyphenol	Hexadecane	34-52	Batch, 350 °C, 28 bar, 1000 rpm, and 7 h	For phenol, benzene (36%), methylcylohexane (6%) and cyclohexylbenzene (43%)	27
35	NiMoW	Guaiacol	-	99	Continuous, 400 °C, 28 bar, and WHSV = 2.7 h^{-1}	Phenol (45%), creosol (15%), catechol (10%), and hydrocarbon (30%)	28
36	CoMoS	p-cresol	Dodecane	78.8-98.7	Batch, 350 °C, 28 bar, 900 rpm, and 7 h	For CoMo-0.5-200, methylcyclohexane (6.3%), methylcyclohexene (1.5%) and toluene (92.2%)	29
37	Ni-WMoS	p-cresol	Dodecane	85-97.9	Batch, 300 °C, 40 bar, 700 rpm, and 6 h	For W-Mo-0.5, methylcyclohexane (66.7%), methylcyclohexene (3.2%) and toluene (30.3%)	30
38	NiMo(W)S	4-methylphenol	Decalin	93.9-97.8	Batch, 300 °C, 30 bar, 800 rpm, and 5 h	Toluene (87.2%), methylcyclohexane (11.3%), and 4-methylcyclohexene (1.5%)	31
39	NiMoWS	4-methylphenol	Decalin	87-100	Batch, 300 °C, 30 bar, 800 rpm, and 5 h	Toluene (95.6%), methylcyclohexane (2.9%), and 4-methylcyclohexene (1.5%)	32
40	MoP, MoS_2, and MoO_x	4-methylphenol	Decalin	30-100	Batch, 300 °C, 30 bar, 800 rpm, and 5 h	See ref[33]	33
41	MoS_2 (effect of adding CTAB)	p-cresol	Dodecane	42-100	Batch, 275 or 300 °C, 40 bar, 900 rpm, and 5 h	See ref[34]	34
42	NiMoS	p-cresol	Dodecane	67-100	Batch, 300 °C, 40 bar, 900 rpm, and 5 h	For NiMo-0.3, methylcyclohexane (67.1%), 3-methylcyclohexene (4.12%), and toluene (28.8%)	35
43	Fe-MoS_2	p-cresol	Dodecane	63.3-98.3	Batch, 250 °C, 40 bar, 900 rpm, and 5 h	For FeMo-0.3, methylcyclohexane (3.7%), methylcyclohexene (1.6%), and toluene (94.7%)	36
44	MoS_2/Al_2O_3	Propylguaiacol	Dodecane	99	Batch, 300 °C, 50 bar, 1000 rpm, and 5 h	Propyphenol (17.5%), cycloalkanes (70.2%) and aromatics (11.8%)	This work
45	$Ni/MoS_2/Al_2O_3$	Propylguaiacol	Dodecane	99	Batch, 300 °C, 50 bar, 1000 rpm, and 5 h	Cycloalkanes (93.9%) and aromatics (7.9%)	This work
46	$Cu/MoS_2/Al_2O_3$	Propylguaiacol	Dodecane	99	Batch, 300 °C, 50 bar, 1000 rpm, and 5 h	Propyphenol (47.2%), cycloalkanes (44.4%) and aromatics (5.7%)	This work

47	$Zn/MoS_2/Al_2O_3$	Propylguaiacol	Dodecane	99	Batch, 300 °C, 50 bar, 1000 rpm, and 5 h	Propyphenol (11.3%), cycloalkanes (67.2%) and aromatics (18.9%)	This work
48	$Fe/MoS_2/Al_2O_3$	Propylguaiacol	Dodecane	99	Batch, 300 °C, 50 bar, 1000 rpm, and 5 h	Propyphenol (26.2%), cycloalkanes (58.1%) and aromatics (16.2%)	This work

a)



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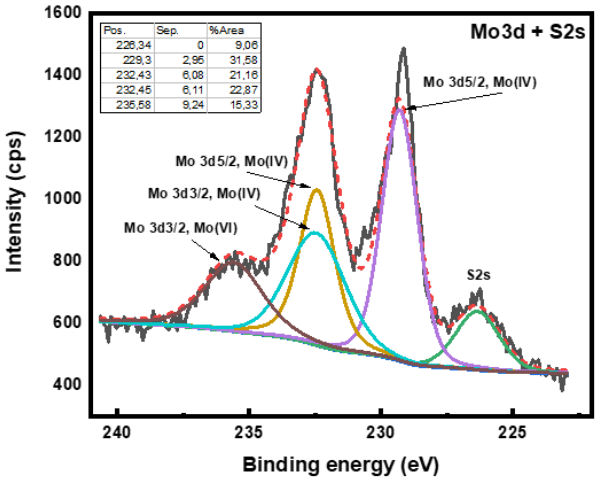
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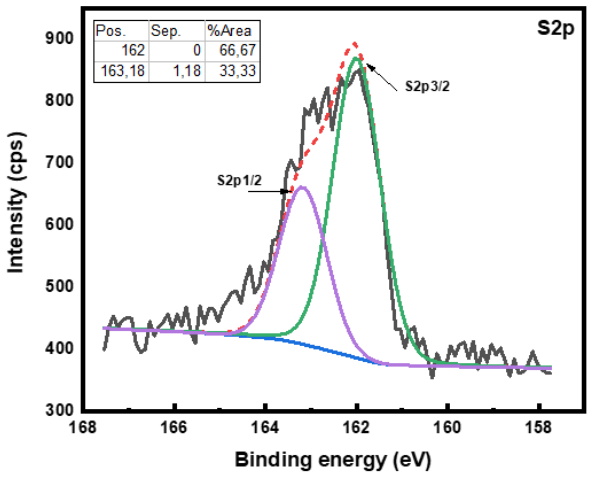
235,58 9,24 15,33
Mo 3d5/2, Mo(IV)
Mo 3d5/2, Mo(IV)
Mo 3d3/2, Mo(IV)
Mo 3d3/2, Mo(VI)
S2s
Intensity (cps)
Binding energy (eV)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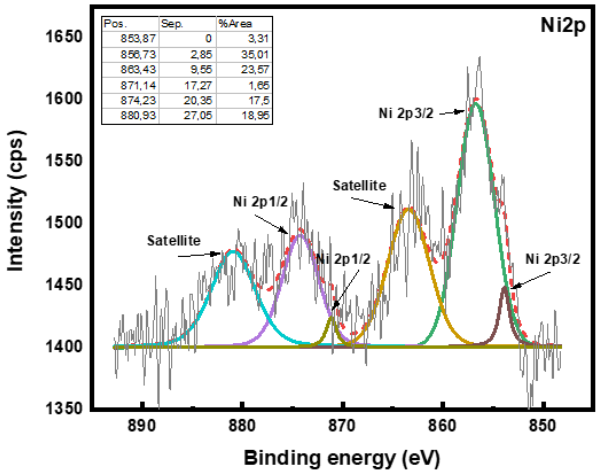
S2p3/2
S2p1/2
Intensity (cps)
Binding energy (eV)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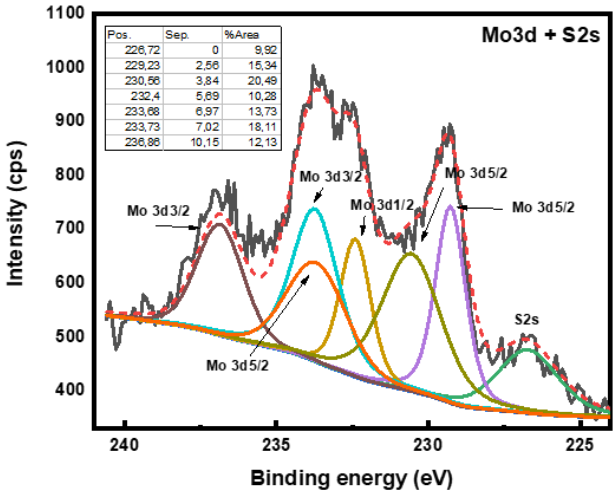
863,43 9,55 23,57
871,14 17,27 1,65
874,23 20,35 17,5
880,93 27,05 18,96
Ni 2p3/2
Satellite
Ni 2p1/2
Satellite
Ni 2p1/2
Ni 2p3/2
Intensity (cps)
Binding energy (eV)

c)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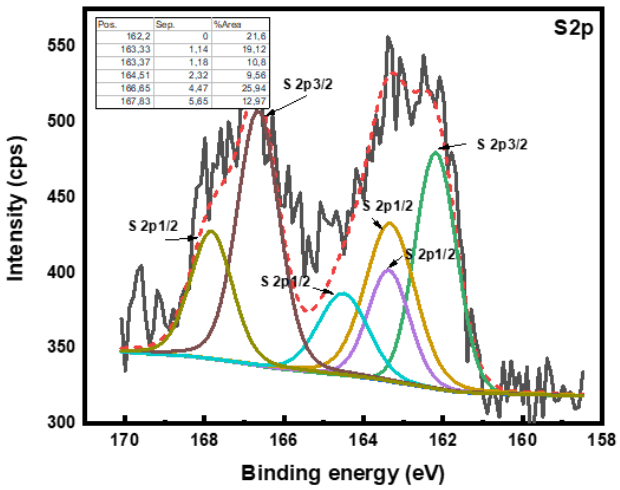
232,4 5,69 10,28
233,68 6,97 13,73
233,73 7,02 18,11
236,86 10,15 12,13
Mo 3d3/2
Mo 3d3/2
Mo 3d1/2
Mo 3d5/2
Mo 3d5/2
Mo 3d5/2
S2s
Intensity (cps)
Binding energy (eV)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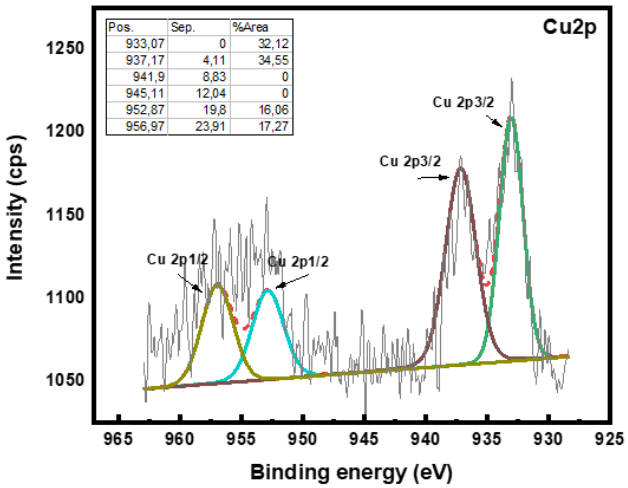
164,51 2,32 9,56
166,65 4,47 25,94
167,83 5,65 12,97
S 2p3/2
S 2p3/2
S 2p1/2
S 2p1/2
S 2p1/2
Intensity (cps)
Binding energy (eV)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941,9 8,83 0
945,11 12,04 0
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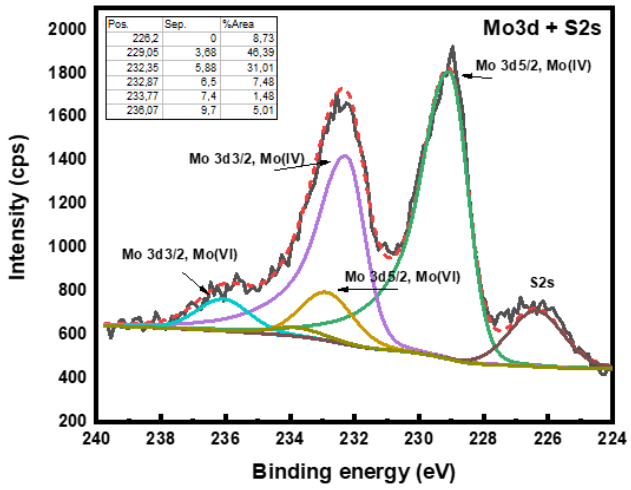
956,97 23,91 17,27
Cu 2p3/2
Cu 2p3/2
Cu 2p1/2
Cu 2p1/2
Intensity (cps)
Binding energy (eV)

d)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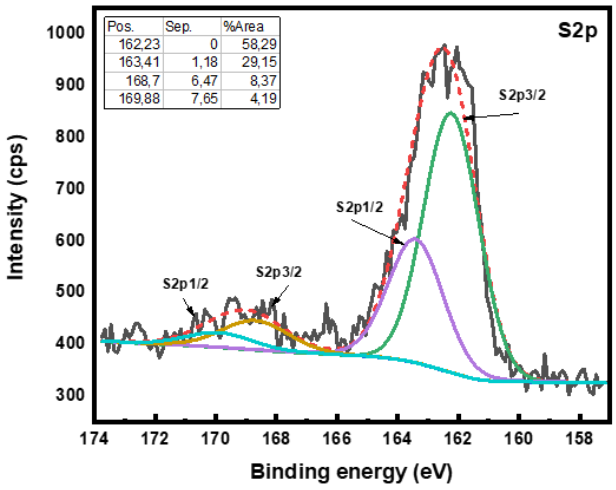
232,87 6,5 7,48
233,77 7,4 1,48
236,07 9,7 5,01
Mo 3d5/2, Mo(IV)
Mo 3d3/2, Mo(IV)
Mo 3d3/2, Mo(VI)
Mo 3d5/2, Mo(VI)
S2s
Intensity (cps)
Binding energy (eV)


S2p
Pos. Sep. %Area
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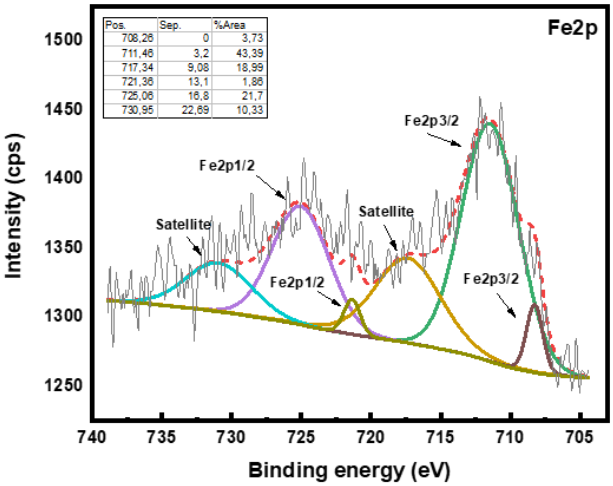
163,41 1,18 29,15
168,7 6,47 8,37
169,88 7,65 4,19
S2p3/2
S2p1/2
S2p1/2
S2p3/2
Intensity (cps)
Binding energy (eV)


Fe2p
Pos. Sep. %Area
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717,34 9,08 18,99
721,36 13,1 1,86
725,06 16,8 21,7
730,95 22,69 10,33
Fe2p3/2
Fe2p1/2
Satellite
Satellite
Fe2p1/2
Fe2p3/2
Intensity (cps)
Binding energy (eV)

e)

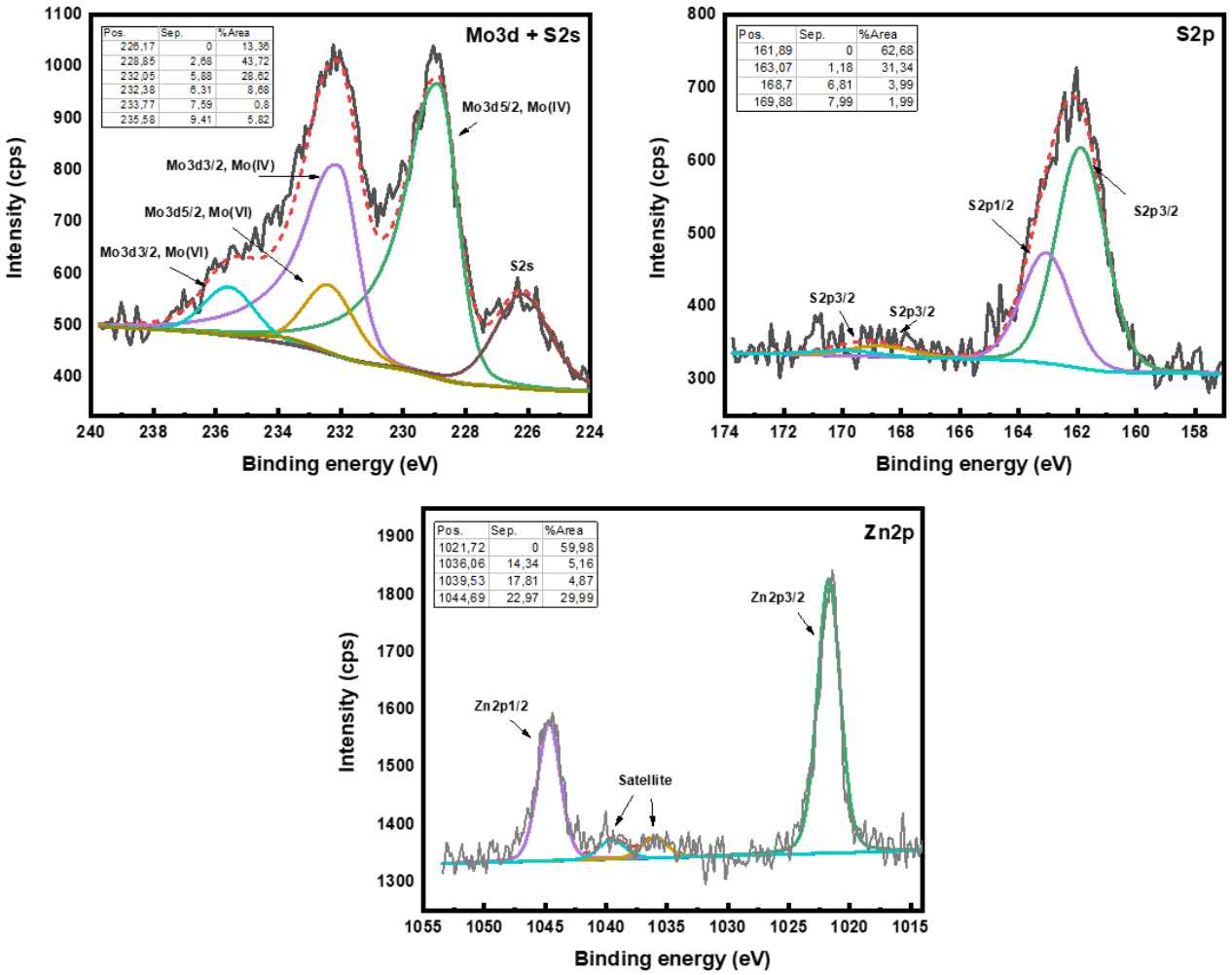

Figure S1 XPS spectra for sulfided a) Mo b) NiMo c) CuMo d) FeMo and e) ZnMo sulfided catalysts.

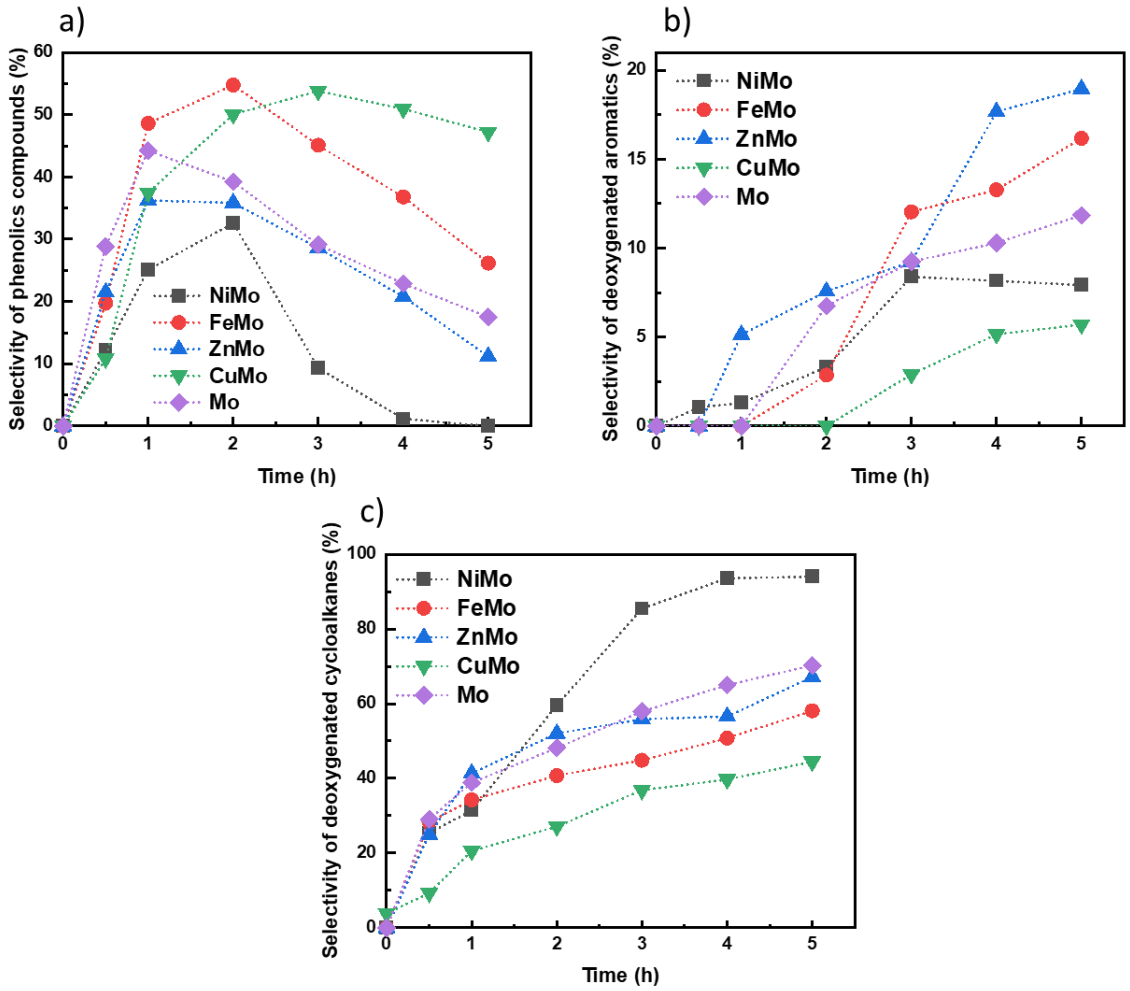

Figure S2 Products selectivity for a) phenolics, b) deoxygenated aromatics, and c) deoxygenated cycloalkanes over sulfided catalysts in HDO of PG.

Table S2: Individual reaction product selectivity (major and side products) over sulfided catalysts in HDO of PG at 5 h.

Selectivity	Mo	NiMo	CuMo	ZnMo	FeMo
Deoxygenated cycloalkanes					
Propylcyclopentane	-	0.8	-	-	-
1-methyl-2-propylcyclopentane	4.5	2.9	3.9	-	4.5
Propylcyclohexane	41.2	80.3	27.6	55.7	49.6
Propylcyclohexene	8.3	-	8.9	-	0.5
1-methyl-2-propylcyclohexane	16.3	10.1	3.0	11.8	3.4
1-butenylcyclopentane	-	-	0.9	-	-
Deoxygenated aromatics					
Propylbenzene	3.6	5.2	3.2	19.1	16.2
3-methyl-6-propylbenzene	-	-	1.2	-	-
1-methyl-3-propylbenzene	8.3	1.6	1.4	-	-
Phenolics					
Propylphenol	12.7	-	41.6	9.6	23.7
4-(1-methylpropyl)-phenol	4.8	-	5.6	1.6	2.1
1-methoxyl-4-(1-methylpropyl)benzene	-	-	0.3	-	-

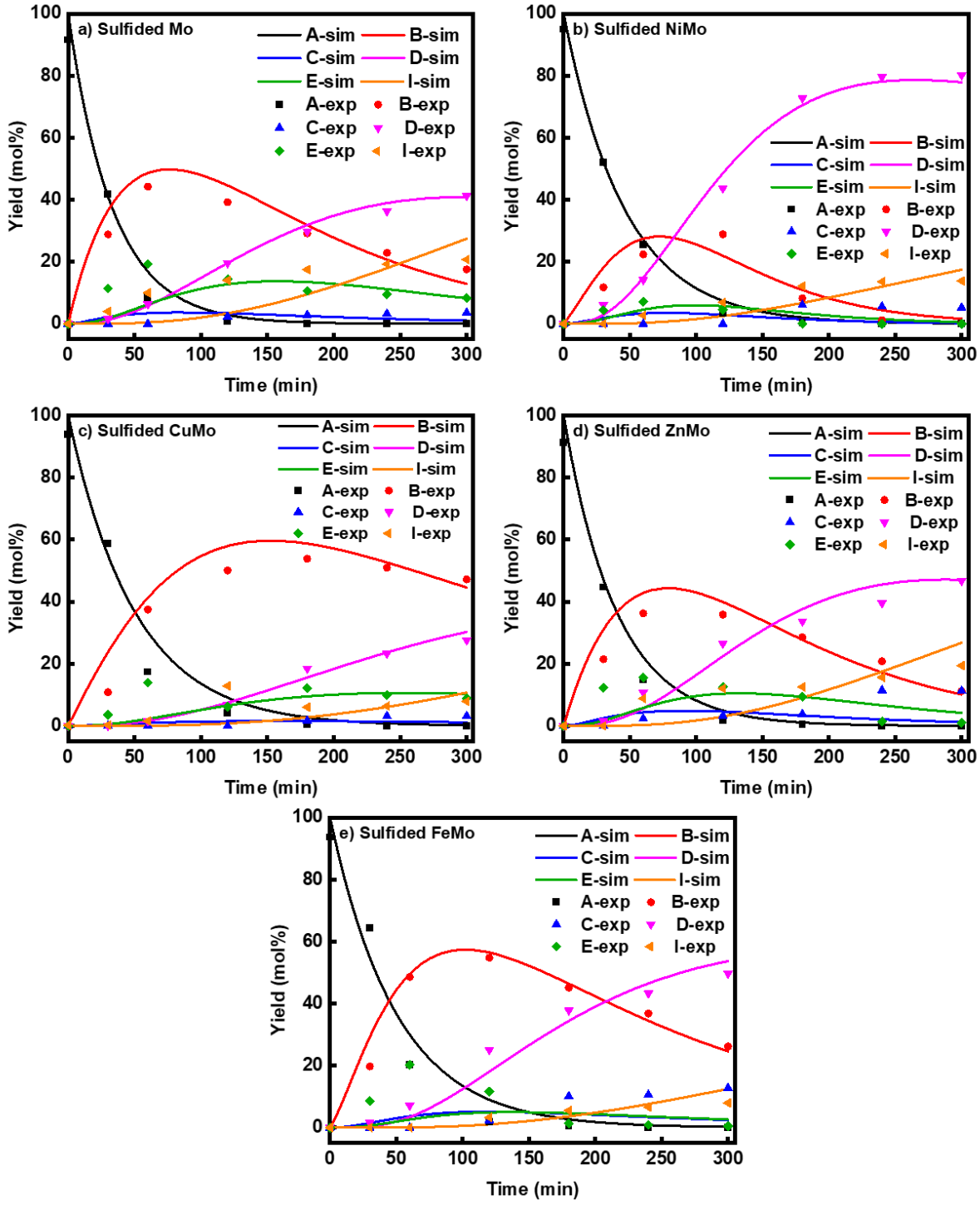

Figure S3 HDO of PG over sulfided (a) Mo, (b) NiMo, (c) CuMo, (d) ZnMo, and (e) FeMo at a total pressure of 50 bar and 300 °C. The solid line denotes the modeling results and the symbols correspond to the experimental values (product yield versus reaction time). Notation: A = Propylguaiacol, B = 4-propylphenol, C = Propylbenzene, D = Propylcyclohexane, E = Propylcyclohexene and I = sum of propylcyclopentane, 1-methyl-2-propylcyclopentane and 1-methyl-2-propylcyclohexane.

*Table S3: Apparent kinetic rate constant values for all catalyst systems for HDO of PG. *Simplified model*

The kinetic rate constant (min^{-1})	***NiMo**	**Mo**	**NiMo**	**CuMo**	**ZnMo**	**FeMo**
k_1	0.0161 ± 0.0036	0.0186 ± 0.0066	0.0053 ± 0.0066	0.0074 ± 0.0058	0.0148 ± 0.0067	0.0071 ± 0.0235
k_2	0.0167 ± 0.0051	0.0089 ± 0.0013	0.0236 ± 0.0039	0.0032 ± 0.0005	0.0105 ± 0.0017	0.0061 ± 0.0008
k_3	0.0597 ± 0.0577	0.0365 ± 0.0808	0.0850 ± 0.2364	0.0575 ± 0.2363	0.0165 ± 0.1105	8.2378×10^{-6} ± 0.4095
k_4	0	0.0063 ± 0.0061	0.0145 ± 0.0059	0.0079 ± 0.0049	0.0074 ± 0.0068	0.0111 ± 0.0242
k_5	0	0.0170 ± 0.0232	0.0288 ± 0.0155	0.0165 ± 0.0141	0.0202 ± 0.0251	0.0610 ± 0.1408
k_6	0	0.0047 ± 0.0057	0.0018 ± 0.0054	0.0054 ± 0.0059	0.0028 ± 0.0044	0.0017 ± 0.0063
k_7	0	0.0156 ± 0.0267	0.0266 ± 0.1015	0.0205 ± 0.0296	0.0113 ± 0.0249	0.0224 ± 0.1075
k_8	0	0.0126 ± 0.0196	0.0035 ± 0.0075	0.0034 ± 0.0248	0.0103 ± 0.0137	0.0034 ± 0.0068
k_9	0	0.0714 ± 0.1204	0.0992 ± 0.2610	0.0941 ± 0.3544	0.0673 ± 0.1333	0.0637 ± 0.4129
k_{10}	0	0.0147 ± 0.0163	0.0553 ± 0.1364	0.0092 ± 0.0234	0.0282 ± 0.0505	0.0644 ± 0.4306
k_{11}	0	0.0039 ± 0.0011	0.0011 ± 0.0002	0.0012 ± 0.0020	0.0033 ± 0.0009	0.0016 ± 0.0009
R_2	90.5	89.7	97.0	90.1	88.3	91.0

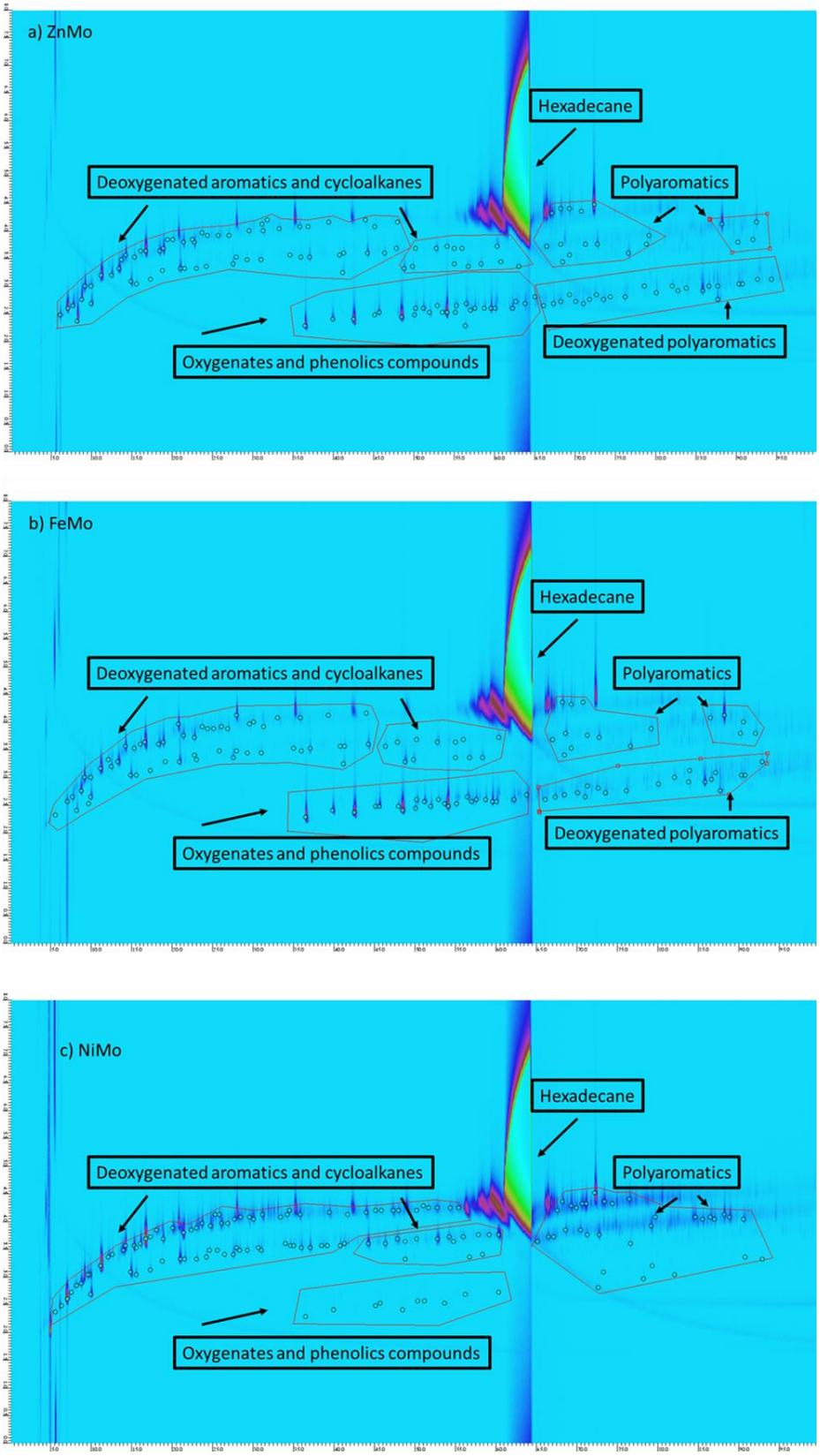

Figure S4 2D GC x GC chromatograms for hydrotreatment of kraft lignin over sulfided a) ZnMo, b) FeMo, and c) NiMo at 340 °C and 40 bar initial H_2 pressure.

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