Electronic Supplementary Material (ESI) for Catalysis Science & Technology.

Guaiacol hydrodeoxygenation reaction catalyzed by highly dispersed, single layered MoS_2/C

Swathi Mukundan,[a] Muxina Konarova,[a] Luqman Atanda,[a] Qing Ma,[a] and Jorge Beltramini*[a]

[a] Nanomaterials center- AIBN and school of chemical engineering, The University of Queensland, Brisbane, QLD-4072, Australia

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During the catalyst synthesis, a certain amount of SO_2 is formed due to presence of atmospheric oxygen. These SO_2 can be removed upon reducing the catalyst in the presence of H_2 at 450 °C for 3 hours. TPR analysis (Fig. 1) shows that SO_2 starts being released at the room temperature in the presence of H_2 and completely removed at 450 °C. The catalyst after being heat treated in the presence of H_2 has no or negligible SO_2 which is also confirmed by XPS (Fig. 2). Molybdenum oxide reduces to Mo metal at high temperature of about 800 °C. There is no H_2S gas released which means the S atom attached to Mo atoms is very stable and it does not react with H_2 to give H_2S. No change was observed for the oxidation state of Mo.

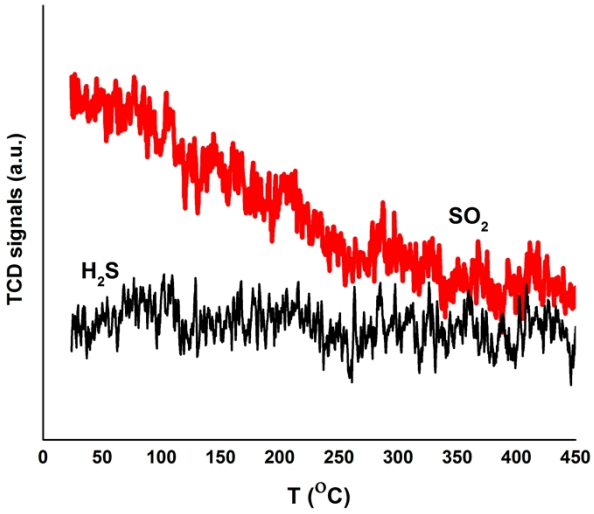

Fig. 1 Temperature programmed reduction (TPR) profile of Fresh MoS_2/C catalyst

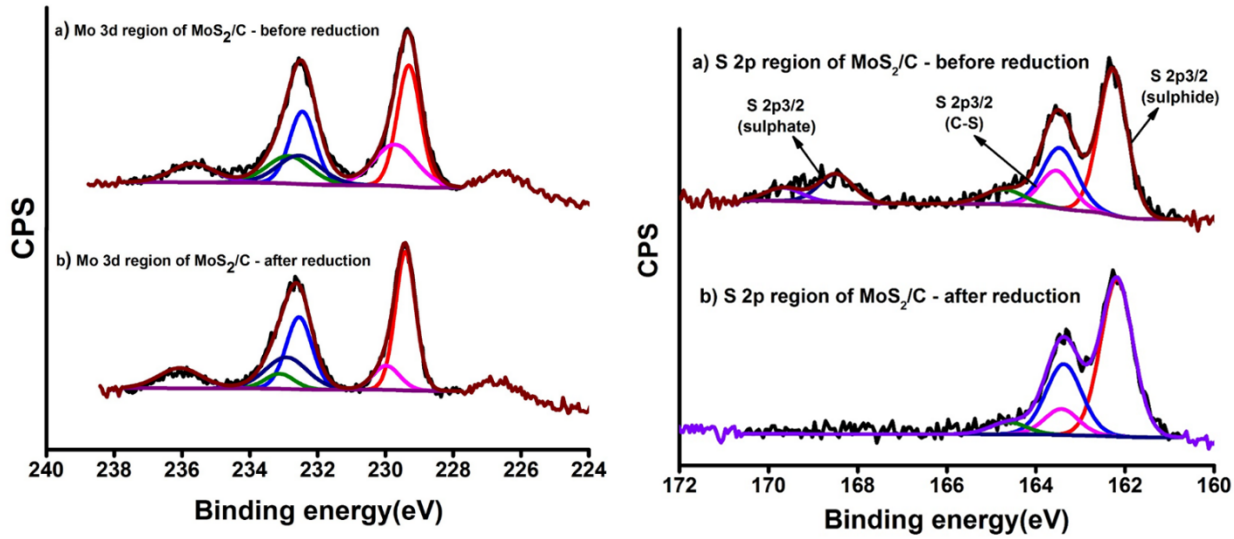

Fig. 2 Curve fitting spectra of (a) Mo 3d region (b) S 2p region of MoS_2/C catalyst before and after reduction

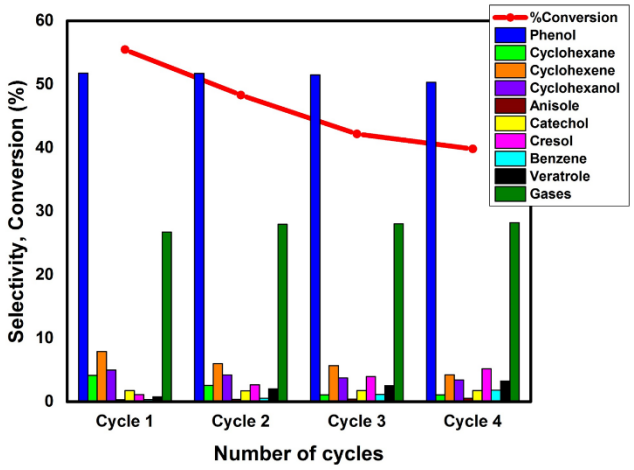

Fig. 3 Catalyst reusability study. Conversion and selectivity of products for HDO of guaiacol over MoS_2/C catalyst

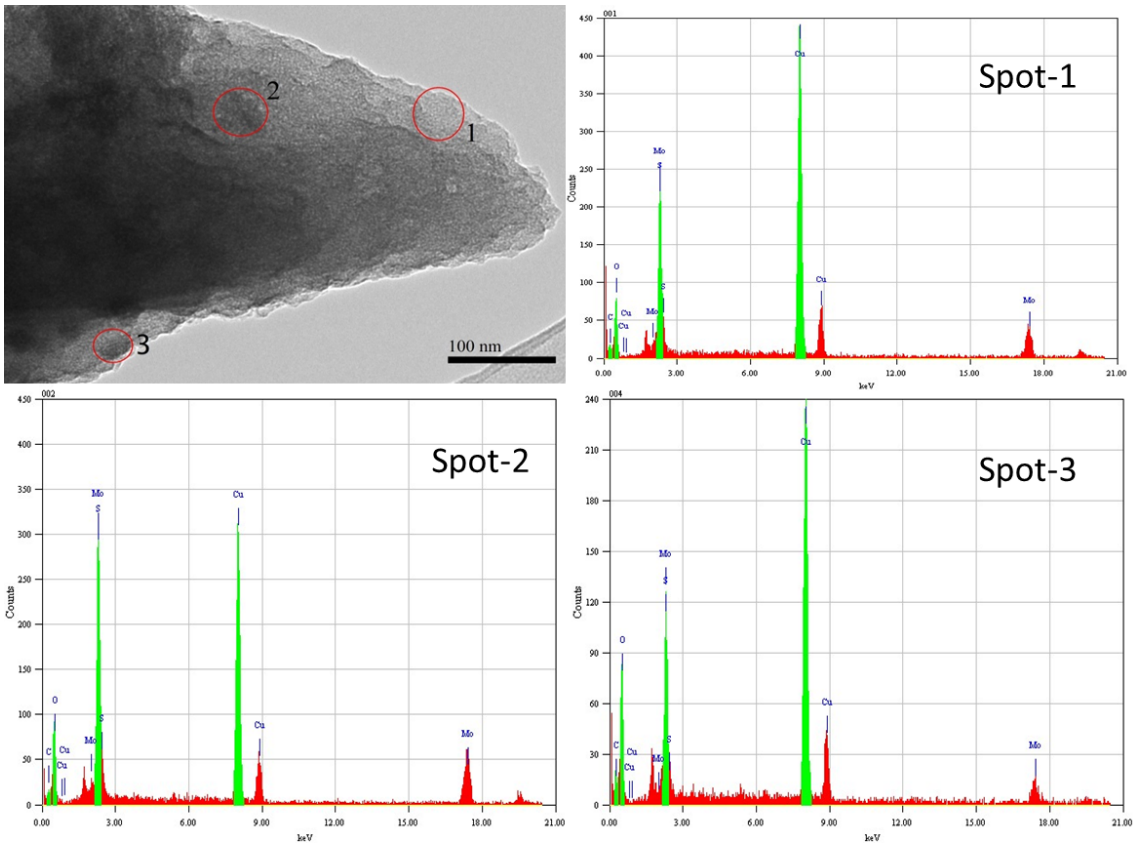

Fig. 4 EDX spectrum of MoS_2/C- fresh at three different spots

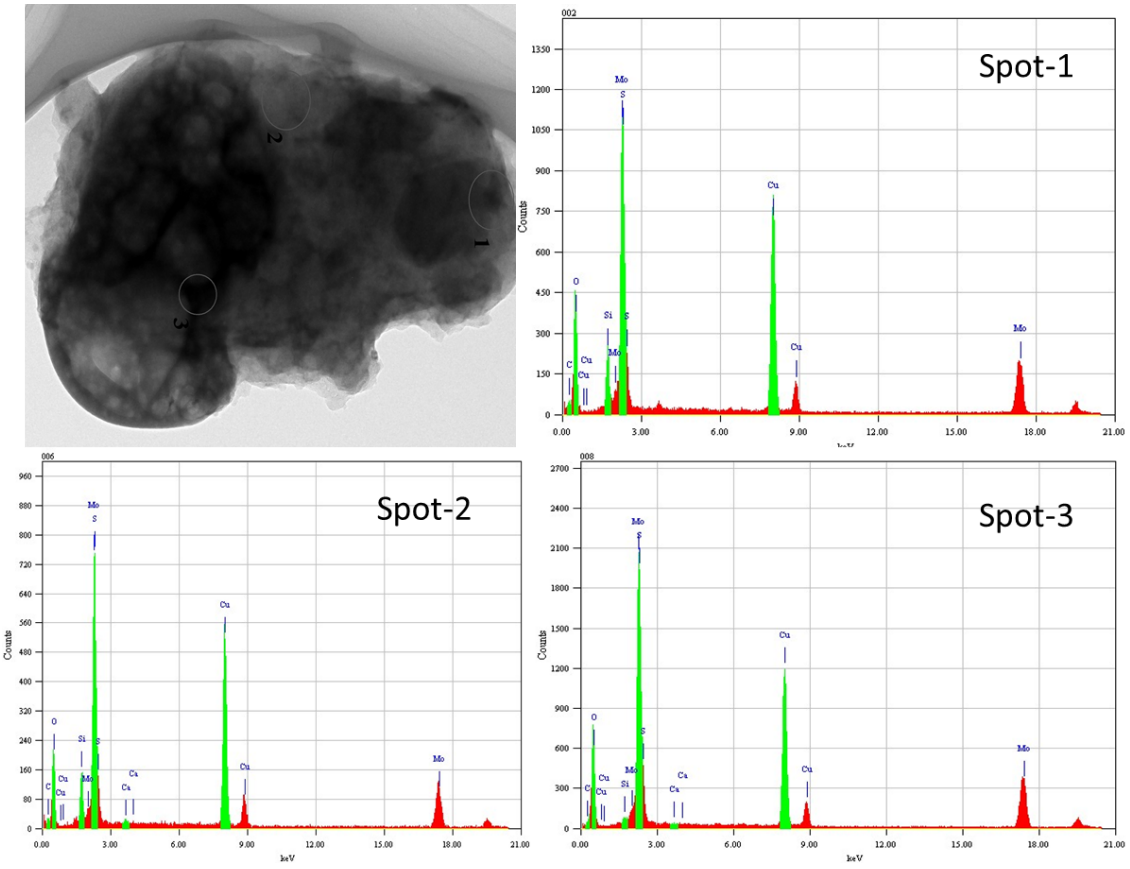

Fig. 5 EDX spectrum of MoS_2/C- cycle 4 catalyst at three different spots

The oxidation state and composition of the elements at the outer surface level of the fresh and used MoS_2/C catalysts were analyzed by XPS. C1s at 284.6eV was used as the reference for interpreting the binding energy of other elements. The general survey scan spectra (Fig. 5) confirms the presence of C, O, Mo and S in the catalyst. C and O were found to be the major components along with Mo and S.

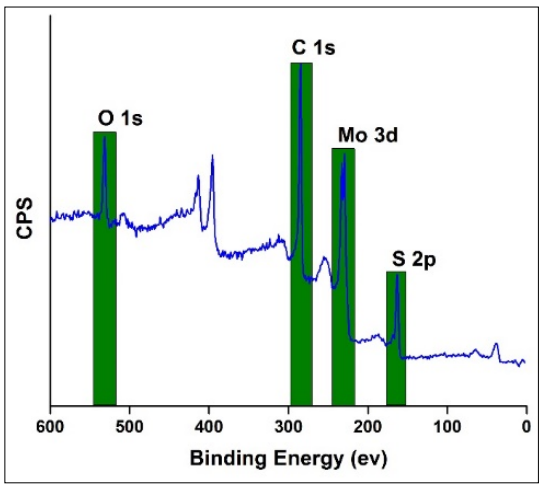

Fig. 6 Survey scan XP spectrum of MoS_2/C- fresh catalyst. CPS= counts s^{-1}

Table 1. Summary of the E^1_{2g} and A_{1g} peak frequencies of fresh MoS_2/C and used catalysts

Catalyst	E^1_{2g} peak frequency (cm^{-1})	A_{1g} peak frequency (cm^{-1})
MoS_2/C- fresh	383.92	403.66
Cycle 1	383.26	403.99
Cycle 2	382.93	406.35
Cycle 3	381.23	405.36
Cycle 4	384.96	407.39

Catalyst MoS_2/C	Binding energy (eV)				
	Mo (+4) $3d_{5/2}$	Mo (+6) $3d_{5/2}$	S $2p_{1/2}$	S $2p_{3/2}$	SO_2
Fresh	229.4	232.6	162.3	164.6	169.8
Cycle 1	229.4	232.6	162.3	164.6	169.4
Cycle 2	229.4	232.6	162.3	164.6	169.7
Cycle 3	229.4	232.6	162.3	164.6	169.7
Cycle 4	229.4	232.6	162.3	164.6	169.3

Table 2. XPS binding energy values (eV) of Mo 3d and S 2p components of fresh and used MoS_2/C catalysts at various oxidation states