Evaluating the performance of MoS_2 based materials for corrosion protection of mild steel in aggressive chloride environment

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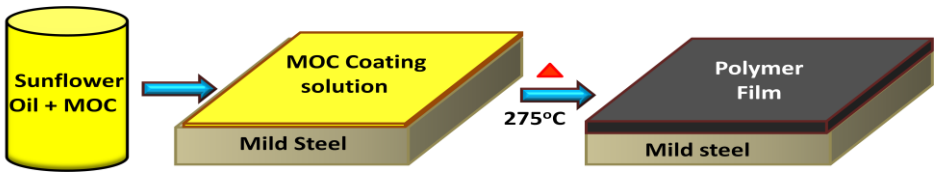
Supporting Information

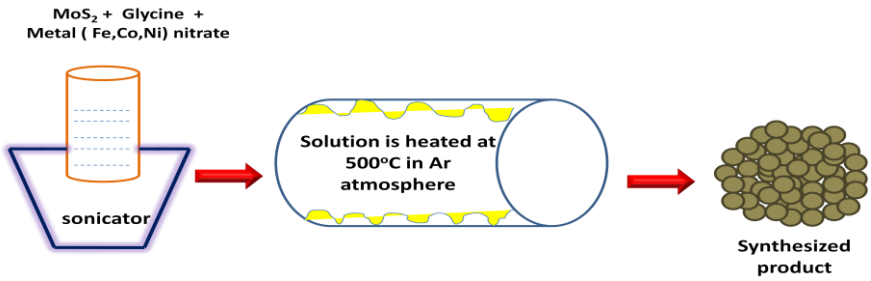
Experimental

Preparation of MoS_2 coated Fe:5 mg of Fe, Co, Ni containing MoS_2nanosheets which is prepared by thermal treatment involving MoS_2 (commercially sourced), glycine, and metal nitrate were taken and is dissolved in 5 ml of sunflower oil. The mixture is then sonicated using a probe-type ultrasonic device to form MoS_2 based coating solution (MOC). The Fe substrate to be coated is cleaned and polished. A calculated quantity of MOC was applied on Fe surface and annealed at 275 °C for 10 min. At this temperature, the triglycerides present in the oil undergo polymerization through oxidation. The reaction involves peroxide formation, decomposition of hydroperoxides, radical formation, and cross-linking. Significant changes in the appearance and the physical and chemical states of the product with respect to the original oil are due to its oxidation. The thickness of the film formed over mild steel is 5-10microns. The corrosion studies were done at room temperature 25 °C.



Scheme 1: Preparation of MOC on Fe by Drop Casting and Heating the Sunflower Oil

Synthesis of Metal containing MoS_2 sheets: The glycine (Gly)–nitrate chemistry previously described for the synthesis of ceramic oxides[1] was adapted to prepare metal containing MoS_2nanosheets. Gly was used to reduce the nitrate ions, resulting in the decomposition of a Gly–nitrate mixture at temperatures around 200 °C.[1]The products of decomposition (e.g. NOx and COx) along with MoS_2 acted as precursors for the synthesis of metal containing MoS_2. In brief, Glycine, metal nitrate (Fe, Co and Ni) andMoS_2 were mixed together in water, followed by tip sonication to form a homogenous aqueous solution. The aqueous mixture was then transferred to a tubular furnace and heated at 500 °C for 2 h under an argon flow to form metal containing MoS_2 sheets.



Scheme 2: Synthesis procedure for metal containing MoS_2 sheets

Instrumentation: X-ray diffraction (XRD) patterns of the samples were measured using Bruker D8 Advance X-ray diffractometer. X-ray photoelectron spectroscopy (XPS) measurement was performed with Sigma probe X-Ray Photoelectron Spectrometer (Thermo VG Scientific) with Al Kα X-ray for excitation. The Field-emission Scanning electron microscopy (FE-SEM) images are done with Carl Zeiss SEM instrument (model number: Supra 55VP/41/46) with an accelerating voltage between 15 kV using SE detector. Energy-dispersive X-ray (EDX) analysis was obtained with an EDX detector installed on the same FE-SEM. A Philips-Tecnai F20 field-emission transmission electron microscopy (FE-TEM) apparatus operated at 200 kV was also used to observe the morphology of as-prepared sample. The samples for TEM measurements were prepared by placing a drop of aqueous dispersion of as-prepared metal containing MoS_2 sheets on carbon-coated copper grids followed by drying. Aberration of the samples was done by using a Taber Abraser model 503 with 500 g loadings. Adhesion test was performed using a cross-cutter, model Erichsen 295/II. Salt spray analysis was carried out with a salt spray test chamber, model Ascott S120T. EIS measurements were carried out on a PAR STAT 2273 Impedance analyzer (Princeton Applied Research) using a conventional three-electrode cell with a platinum counter electrode and a silver/silver chloride reference electrode. For Tafel polarization, the potential of the working electrode was scanned from −0.2 to +0.2 V versus open circuit potential (OCP) at the scan rate of 1 mV/s. From the anodic and cathodic polarization curves, the Tafel regions were identified and extrapolated to corrosion potential (Ecorr) to get the corrosion current (Icorr) by using the auto-Tafel fit feature of Corrview software. All the experiments were carried out under room temperature (25 °C).

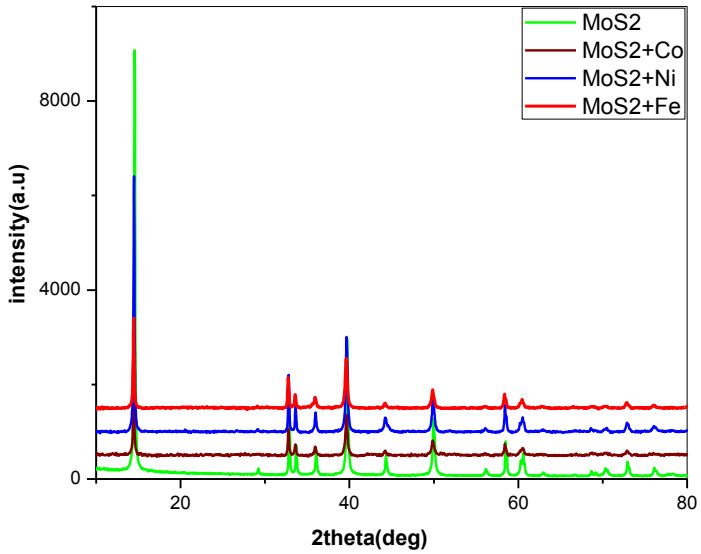


Figure S1a. XRD pattern of pristine MoS2 and metal doped MoS2 powders

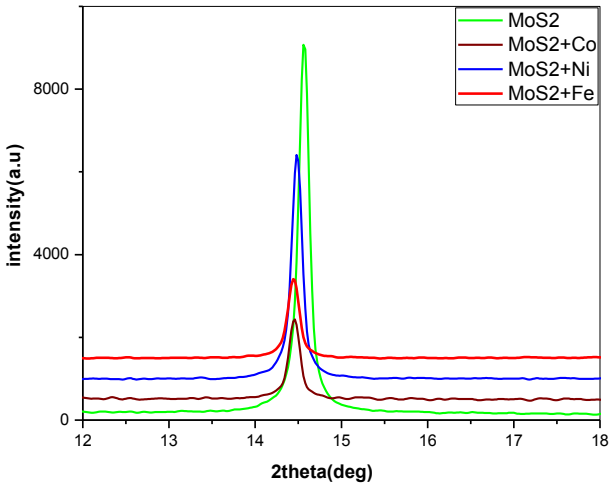


Figure S1b. XRD pattern showing (002) peak

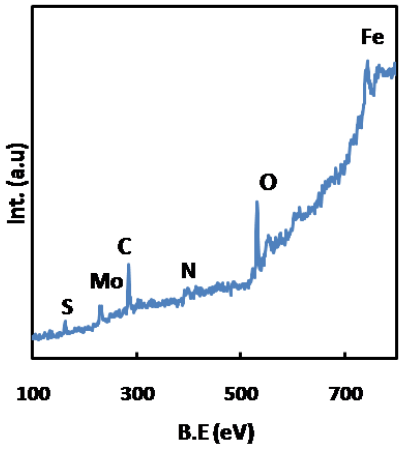


Figure 1c. XPS survey spectra of as-prepared Fe-MoS_2 powder

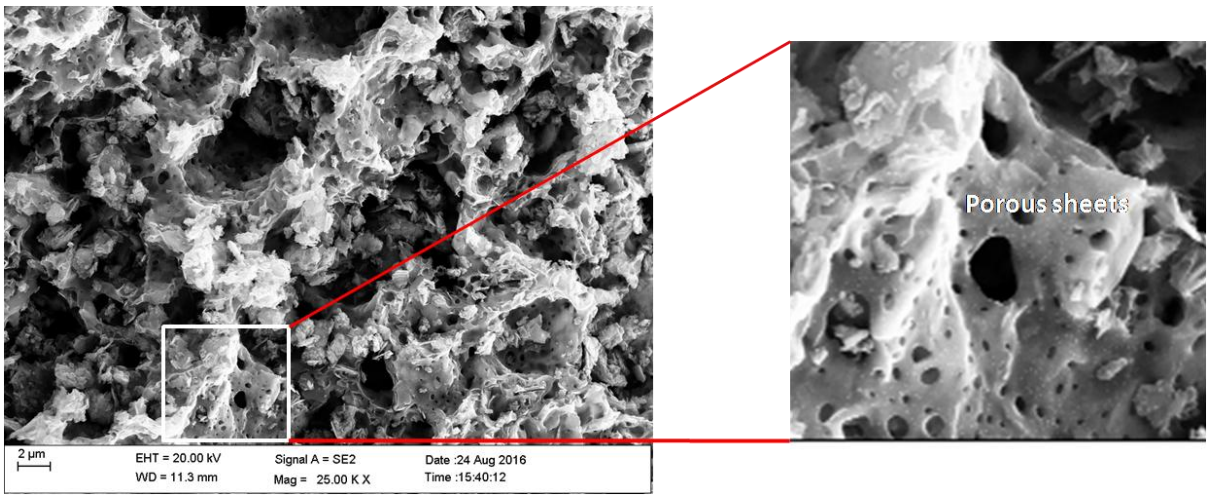


Figure S2 a. FE-SEM image of MoS_2 powder showing the presence of porous sheets. The magnified image shows the presence of large number of pores in the sheet.

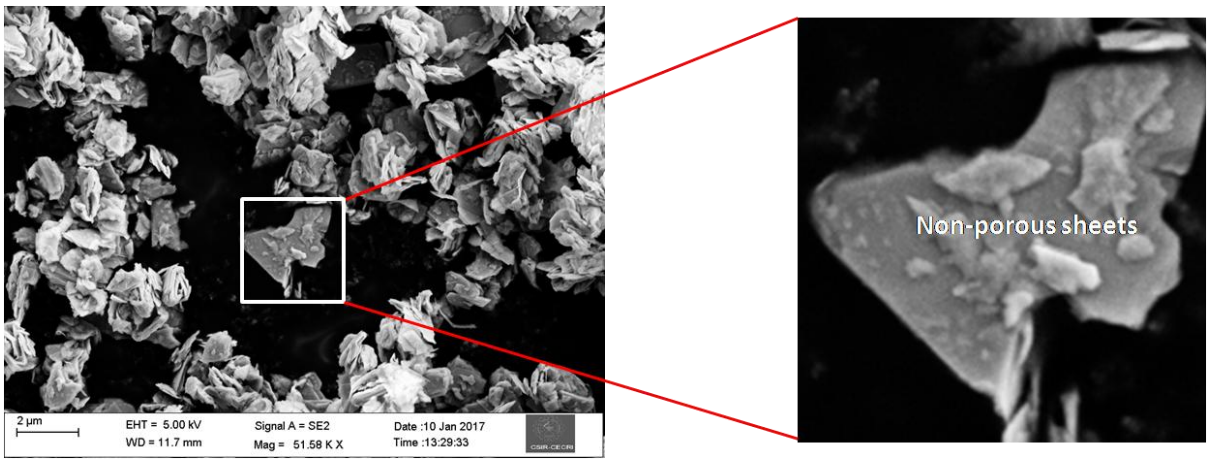


Figure S2 b. FE-SEM image of MoS_2 powder showing non-porous sheet like structures. The magnified image shows the absence of pores in the sheet (non-porous structures)

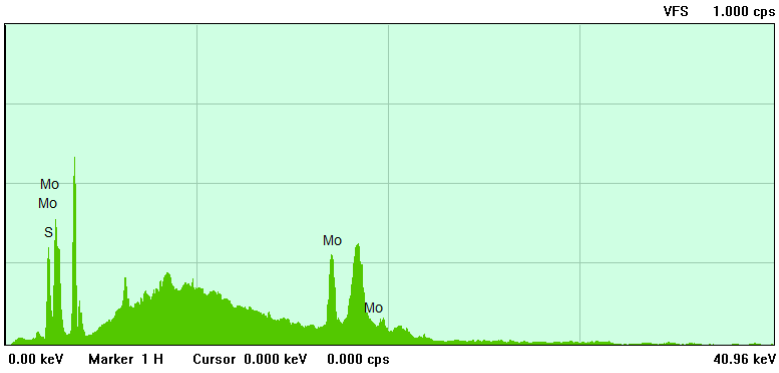


Figure S3 (a). XRF spectra of oil coating with MoS_2

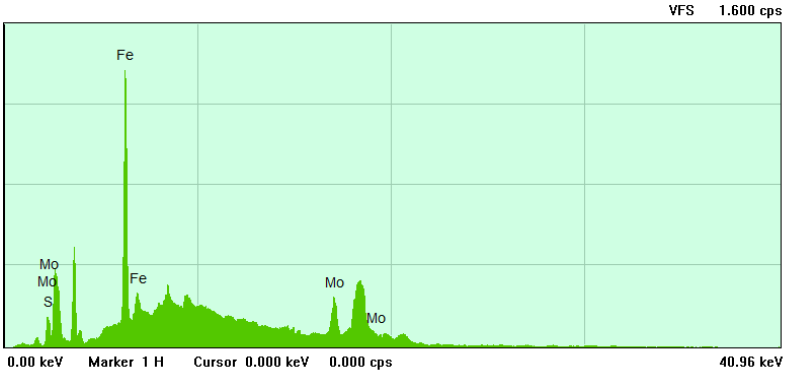


Figure S3 (b). XRF spectra of oil coating with Fe-MoS_2

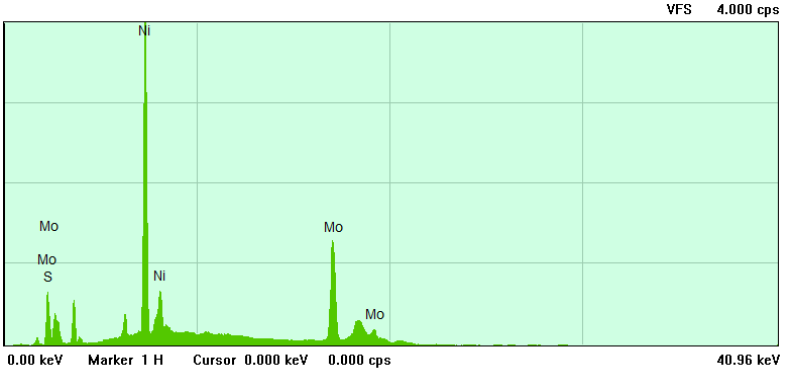


Figure S3 (c). XRF spectra of oil coating with Ni-MoS_2

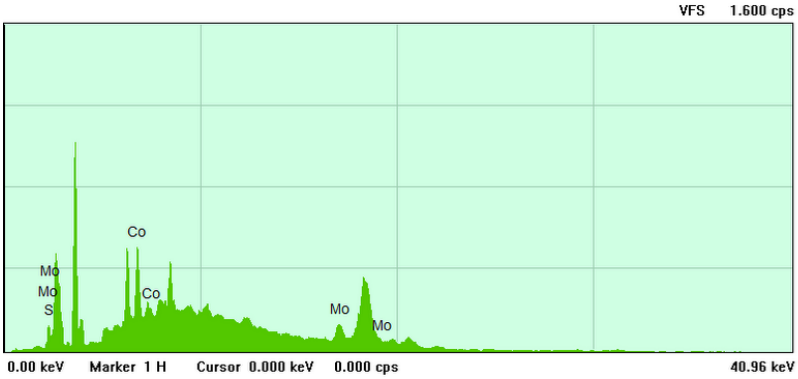


Figure S3 (d). XRF spectra of oil coating with Co-MoS_2

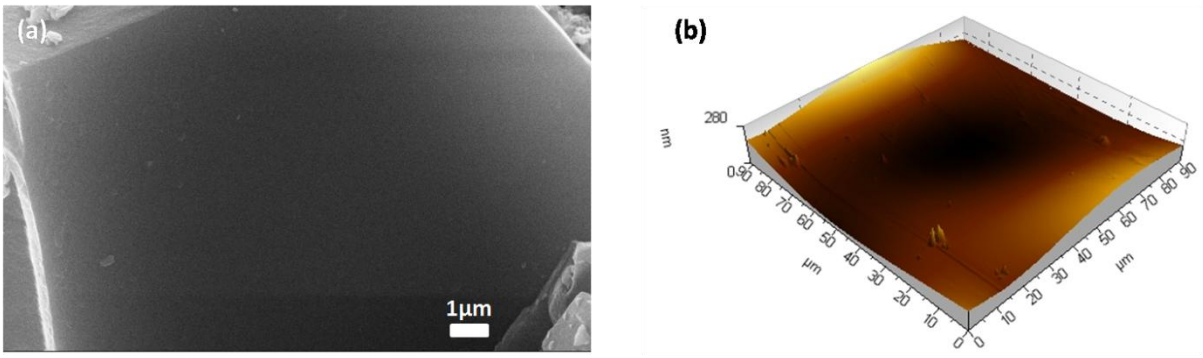


Figure S4 . (a) FE-SEM and AFM (b) images of MOC (MoS_2-Fe) coated on Fe shows smooth morphology without any micro/nanostructures.

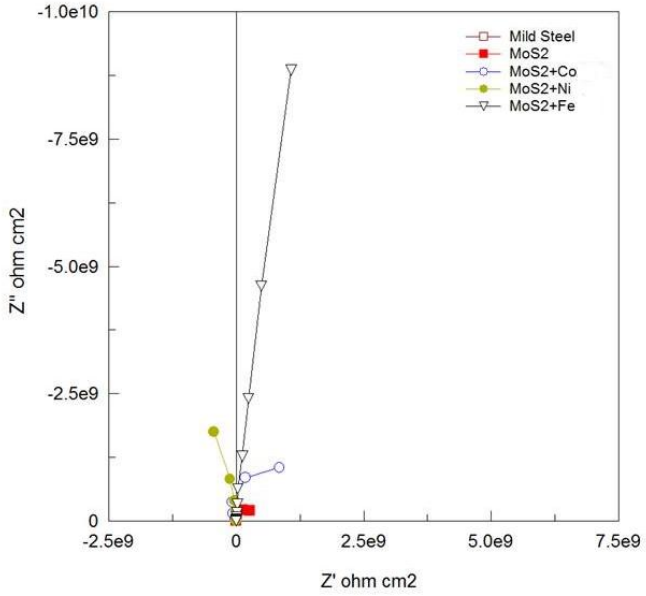


Figure S5: Impedance spectra of MOC

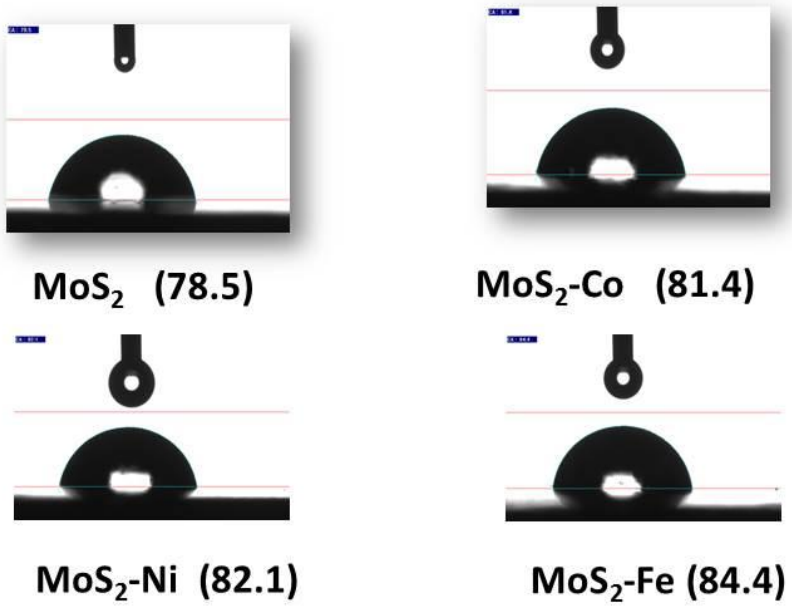


Figure S6: Contact angle of MOC on Fe