Electronic Supplementary Material (ESI) for Catalysis Science and Technology

Synthesis, High Resolution Electron Microscopic Investigation and Catalytic Properties of Molybdenum Disulfide Nanoplates.

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The synthesis of MoS_2 via the milling and reduction sometimes produces products with content of sulfur oxides. We worked with several synthesis and annealing temperatures, as well as several reaction times. The reaction times were 2 hours, 2.5 hours and 3 hours. When the samples were left to react for two hours with heating, the product obtained had presence of sulfur oxides, this phenomenon was avoided increasing the reaction time, the samples synthesized at 2.5 hours and 3 hours do not show oxysulfides.

Figure S1 reveals the XRD pattern of the MoS_2 plates. Figures S1a and S1b show the samples synthesized at 600°C revealing the presence of some peaks corresponding to MoO_3. This leads to the conclusion that the conversion is not complete at this working temperature. In the XRD pattern of the MoS_2 plates synthesized at 700°C (fig. S1c, S1d, S1e and S1f), the peaks observed correspond to the hexagonal phase of MoS_2 (2H-MoS_2). Annealing temperature increases the sharpness and resolution of the peaks, indicating that the crystallinity of the sample increases with the temperature. The reflections of MoS_2 have been indexed to the same polymorph stated on the paper. The XRD pattern shown in figure S1f belongs to the sample synthesized at 700°C and annealed at 1000°C, this also corresponds to the 2H phase of MoS_2. Figures S1g, S1h and 1i show also the XRD pattern of the samples synthesized at 800°C with no annealing, and annealed at 900°C and 1000°C. The patterns correspond to the 2H phase of MoS_2. Here again the sharpness of the peaks increases with higher annealing temperatures, indicating that the crystallinity of the samples increases. The (002) peak of MoS_2 is clearly seen in all the diffraction patterns, except in the spectra shown in figures S1a and S1b, samples synthesized at 600°C, this may be due to an incomplete conversion of the oxide to the corresponding sulfide.

Figure S2a shows the SEM micrograph of the MoS_2 plates synthesized at 600°C and annealed at 900°C. The sample looks amorphous and no hexagonal plates can be seen. Figure S2b shows the SEM micrograph of MoS_2 synthesized at 600°C and annealed at 1000°C. This sample has some hexagonal plates, but still amorphous regions and large rods were observed. The micrographs of MoS_2 synthesized at 700°C with no annealing and annealed at 800°C (fig. S2c and S2d) show amorphous areas due to the non effective annealing. The images of MoS_2 synthesized at 800°C and annealed at 900°C and 1000°C are shown in figures S2e, S2f and S2g, respectively. The samples have some hexagonal plates, but also some rods are present. Hence this temperature of synthesis was also not suitable. The temperature of 700°C followed by annealing at 900°C or 1000°C seems to be the best suited temperature for the synthesis of the MoS_2 plates.

Figures S3a, S3b and S3c show the Raman spectra of the samples synthezied at several conditions. In figures S3a, S3b and S3c are seen the Raman peaks: At 280 and 375 cm^{-1} appear the E_{1g} peak of crystalline MoS_2 and the E^1_{2g}, which is characteristic of hexagonal MoS_2. At 408 cm^{-1} the A_{1g} peak is present. The peaks at 220 and 340 cm^{-1} correspond to J_2 and J_3 peaks and are not normally seen in the Raman spectrum of 2H-MoS_2 crystals, the first one may be due to changes in the electronic state, the second one may be due to the presence of sulfur oxides. Strong peaks are observerd at 805 cm^{-1} and 980 cm^{-1}, they correspond to the presence of an oxysulfide, probable SO_3.

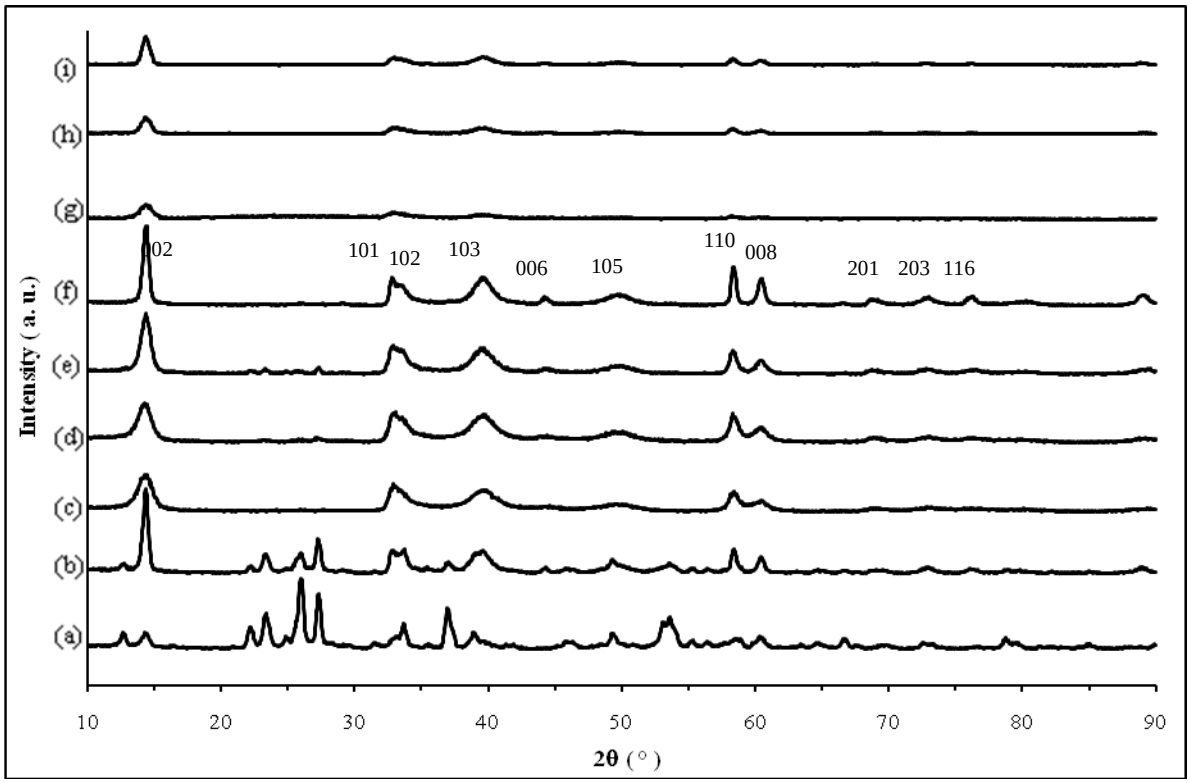

Fig. S1 XRD patterns of the MoS_2 plates synthesized and annealed at different reaction conditions: (a) 600°C-900°C; (b) 600°C-1000°C; (c) 700°C, no annealing; (d) 700°C-800°C; (e) 700°C-900°C; (f) 700°C-1000°C; (g) 800°C, no annealing; (h) 800°C-900°C; (i) 800°C-1000°C. The peaks are indexed and it is shown in above the samples 700-1000°C, which is the more crystalline of the samples.

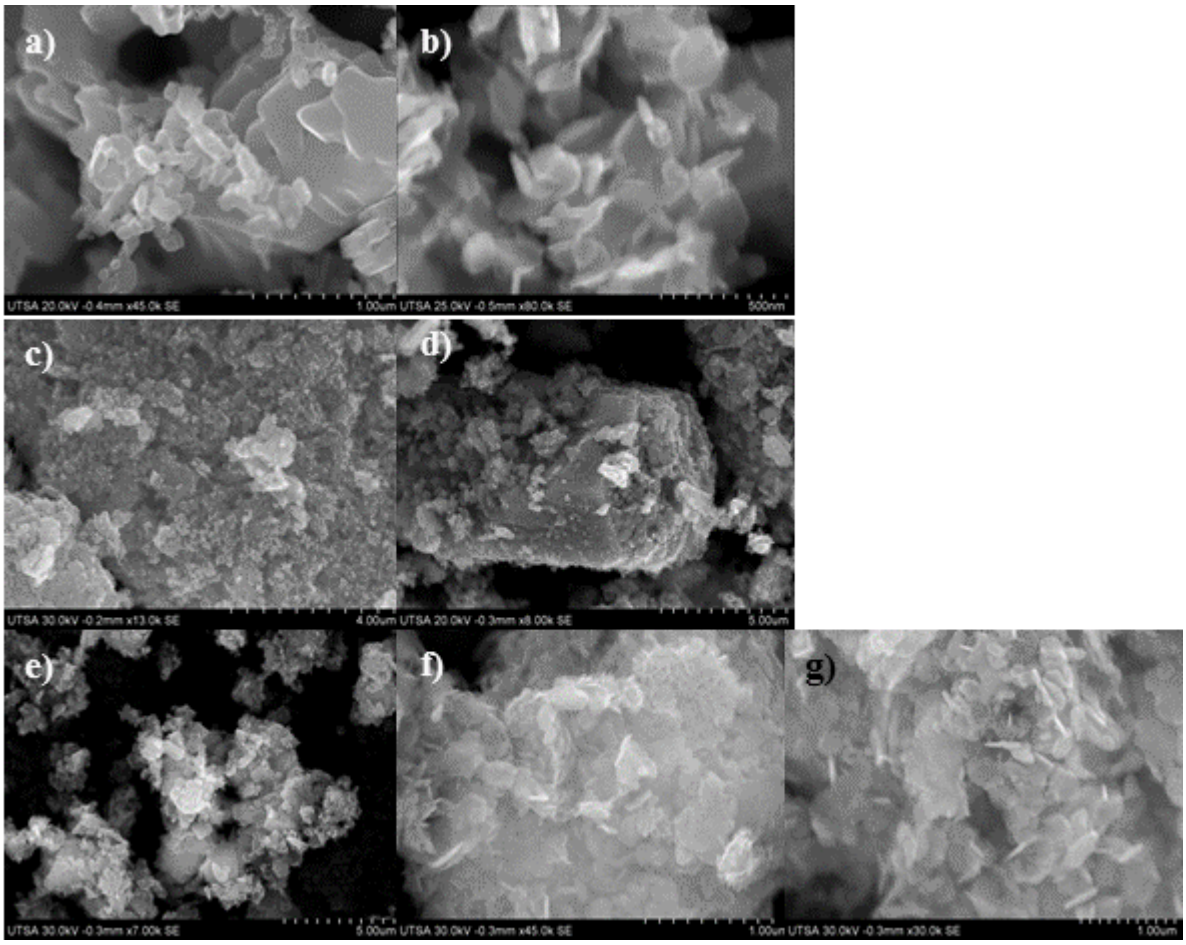

Fig. S2 SEM micrographs of the MoS_2 plates synthesized and annealed at different reaction conditions: a) 600°C-900°C; b) 600°C-1000°C; c) 700°C, no annealing; d) 700°C-800°C; e) 800°C, no annealing; f) 800°C-900°C; g) 800°C-1000°C.

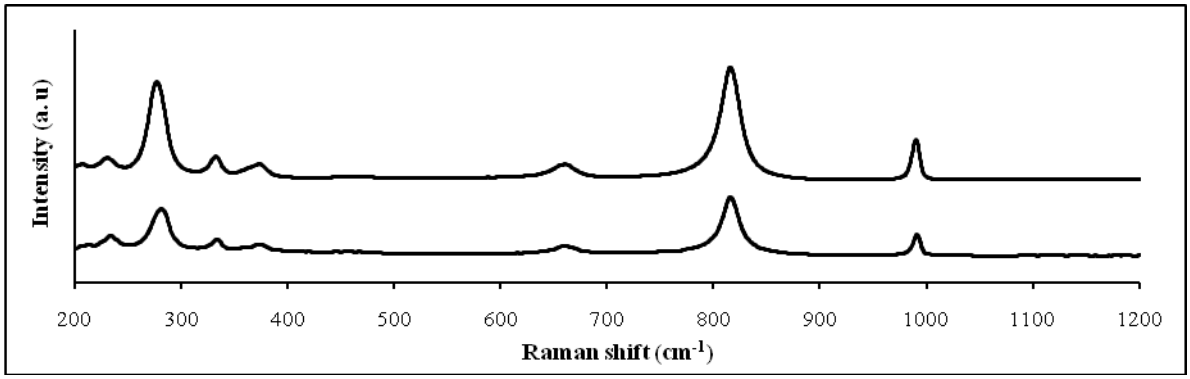

Fig. S3. Raman spectra of the MoS_2 plates synthesized at 600°C and annealed at two different temperatures (900°C and 1000°C).

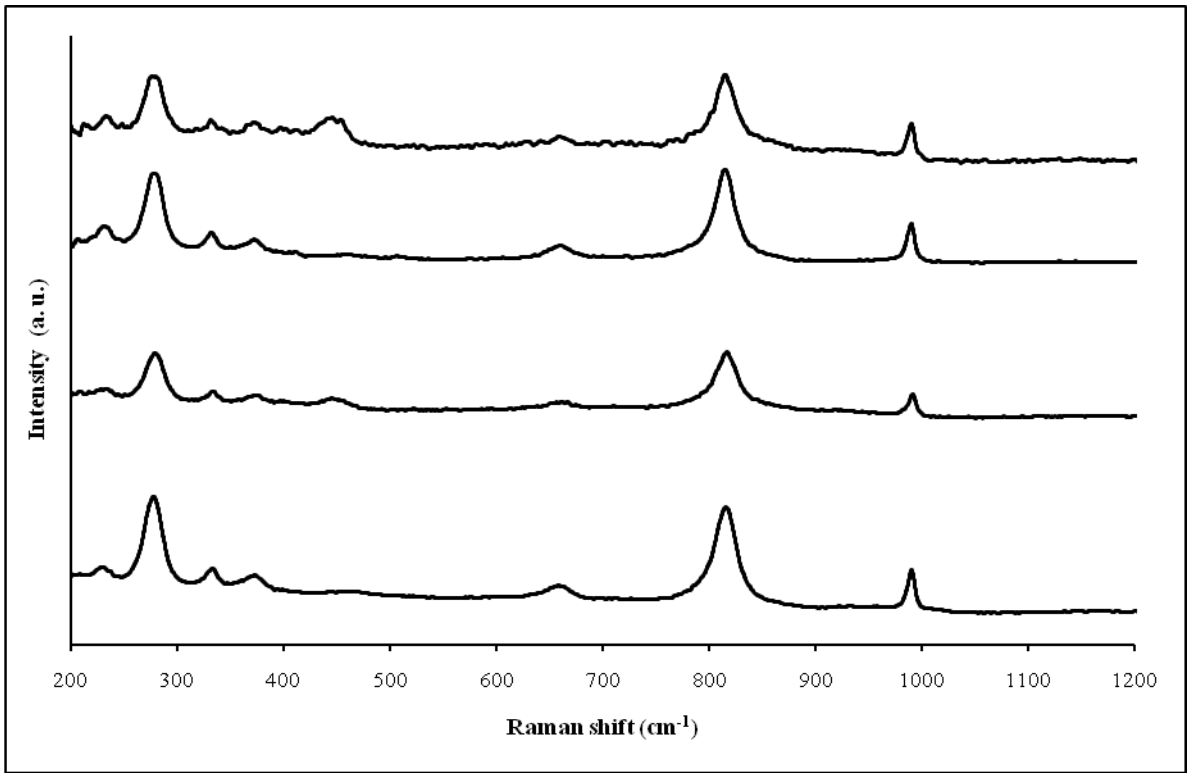

Fig. S3b Raman spectra of the MoS_2 plates synthesized at 700°C and annealed at three different temperatures (800°C, 900°C and 1000°C).

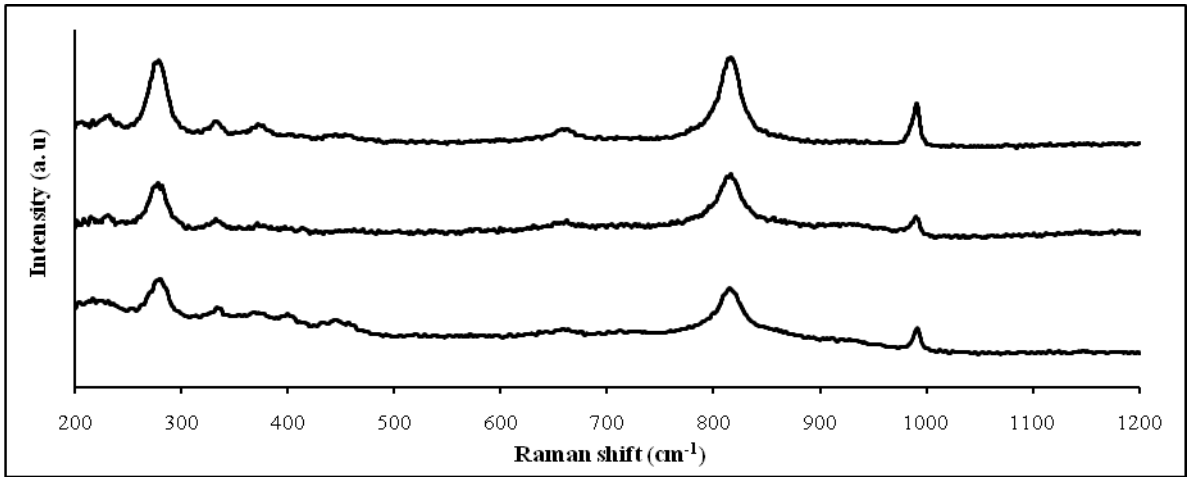

Fig. S3c Raman spectra of the MoS_2 plates synthesized at 800°C and annealed at two different temperatures (900°C and 1000°C).