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In Situ XRD Study of MoO_3 Particles During Heat Treatment and Their Capacity of Removing the MB

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The effects of heat treatment (HT) at different temperatures from 25 to 600 ° C were studied in their morphology and crystal structure for particles of MoO_3 with orthorhombic (OW sample) and hexagonal (HW sample) phases. Employing high energy mechanical milling technique was possible to get nanoparticles with size below 40 nm at 30 min milling time from HW sample with previous calcination process (HWCM sample) [1]. The milling nanoparticles showed greater removal of Methylene blue (MB) compared to the microparticles [2].

The morphology and microstructure were determined using electron microscopy techniques and X-ray diffraction. Figures 1a and 1b show the X-ray diffraction patterns evolution during HT of the OW and HW samples, respectively. In general, at all temperatures the diffraction patterns correspond to the orthorhombic phase (see Fig. 1a) and practically remains stable as temperature increase, however there is a slight decrease in width at 600 ºC, indicating that exist an increase in crystallite size due to atomic diffusion during HT. Figure 1b, shows the X-ray diffraction patterns evolution during HT of the HW sample, above 440°C, hexagonal phase changes into orthorhombic phase, and an apparent broadening of the diffraction peaks is observed, this phenomenon corresponds to a delamination of the hexagonal phase [3]. The secondary electrons SEM image on OW sample before HT shows elongated, thin and smooth surface ribbon-like structures (see fig. 2a). For the 440 ° C temperature some particles were maintained with tape-shaped morphology but with larger dimensions (see fig, 2b). Figure 2c shows the SEM image of secondary electrons obtained before the HT of the HW sample, the particles show hexagonal prism shape morphology. Fig. 2d shows different agglomerates particles showing a micro-lamella type morphology corresponding to the orthorhombic phase of the HWC (before the MM process) sample submitted at 440 ° C. On the other hand an irregular morphology of agglomerated nanoparticles was observed when the particles were subjected to mechanical milling (samples named HWCM) as is shown in the TEM image (see Fig.3a). These particles were also analyzed through SAED diffraction patterns annexed. The nanometric particles produced allowed an increase in surface area, improving noticeable the adsorption activity of MB in a time of 50 min for the calcined milled HWCM sample. The nanometric particles powder increased the speed discoloration, without using photonic irradiation.

References:

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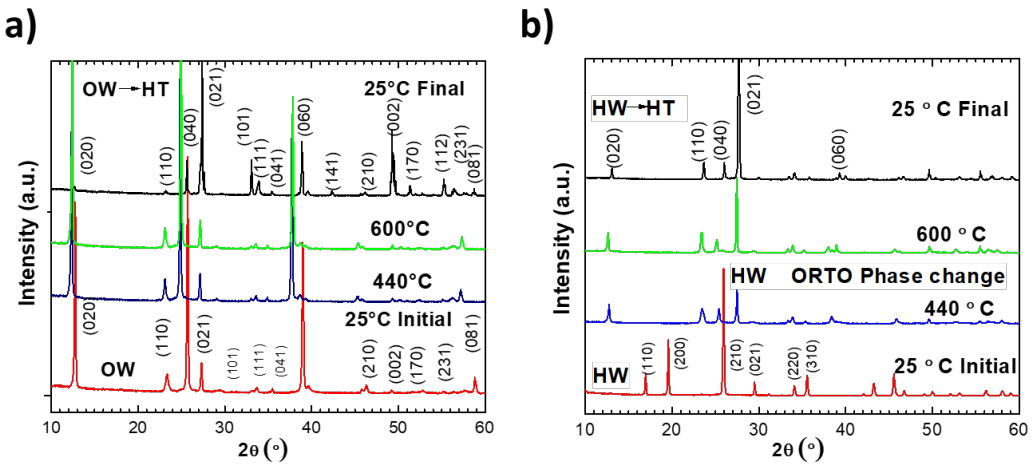

Figure 1. XRD patterns of the microwave synthesis (OW, HW), submitted to heat treatment a) OWTT and b) HWTT.

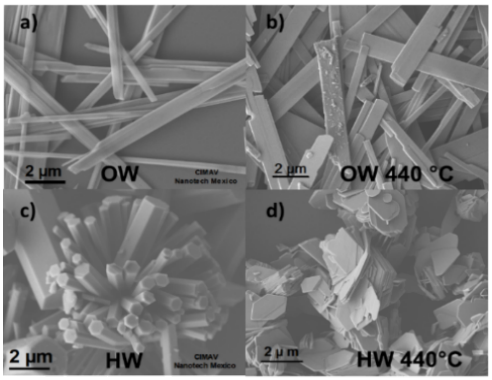

Figure 2. Particle morphology for orthorhombic phase SEM Image before HT, a) OW and c) HW, at 440 °C, b) OW and e) HW.

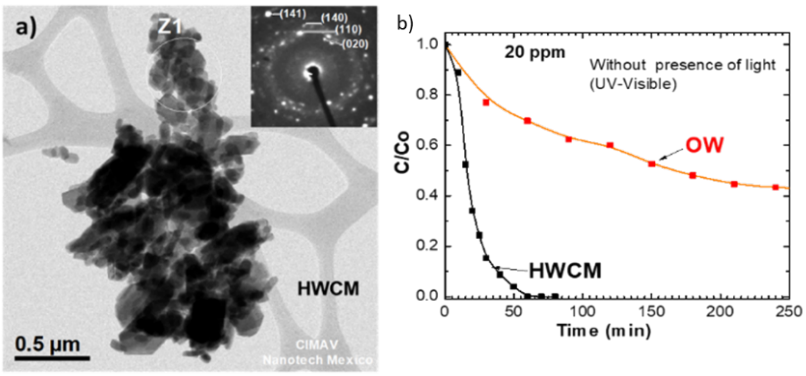

Figure 3. a) TEM image and SAED pattern of HWCM and b) Discoloration process of 20 ppm MB solution in the dark, for samples OW and HWCM.

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