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Micro-structural characterization of inorganic cryptomelane nanomaterials synthesized by use of controlled O_2 and cold aging in acidic conditions

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For the past century researchers have invested great efforts in the synthesis of materials with various shapes including single crystals, wires, helices, rods, tubes, and films from inorganic materials [1-2]. A new friendly green chemistry method was developed to prepare inorganic cryptomelane nanomaterials with the highest surface area ever reported for this class of materials. The synthesis is based on the oxidation of $MnSO_4$ by permanganate ion in acidic conditions in the presence of about 2.5 x 10^{-4} M O_2 at 60^{o}C in a closed reaction vessel and aging at 4 ^{o}C for 12 h. It was found that the aging time and concentration of O_2 affects the size of the crystals formed in the final product. Various microscopy techniques were employed to investigate the microstructure of this material. Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) technique are employed to image this nanomaterial. A fibrous structure with about 100 nm striations is identified by TEM BF (Bright Field) imaging, as seen in Fig. 1 (a). However, in HRTEM mode, this nanomaterial shows a double helices structure lying on (110) plane, Fig. 1 (b). To model the structure, O-XANES(Oxygen X-ray Absorption near edge spectroscopy) was used to identify the phase structure indicating only single phase existed in this materials, as shown in Fig. 2. A geometry model was proposed and the d-spacing of this material was simulated and examined by XRD. The calculated d-spacing based on the proposed model and measured d-spacing by XRD are identical. The inorganic double helices of cryptomelane nanomaterial may be associated with low temperature and controlled oxygen condition.

References

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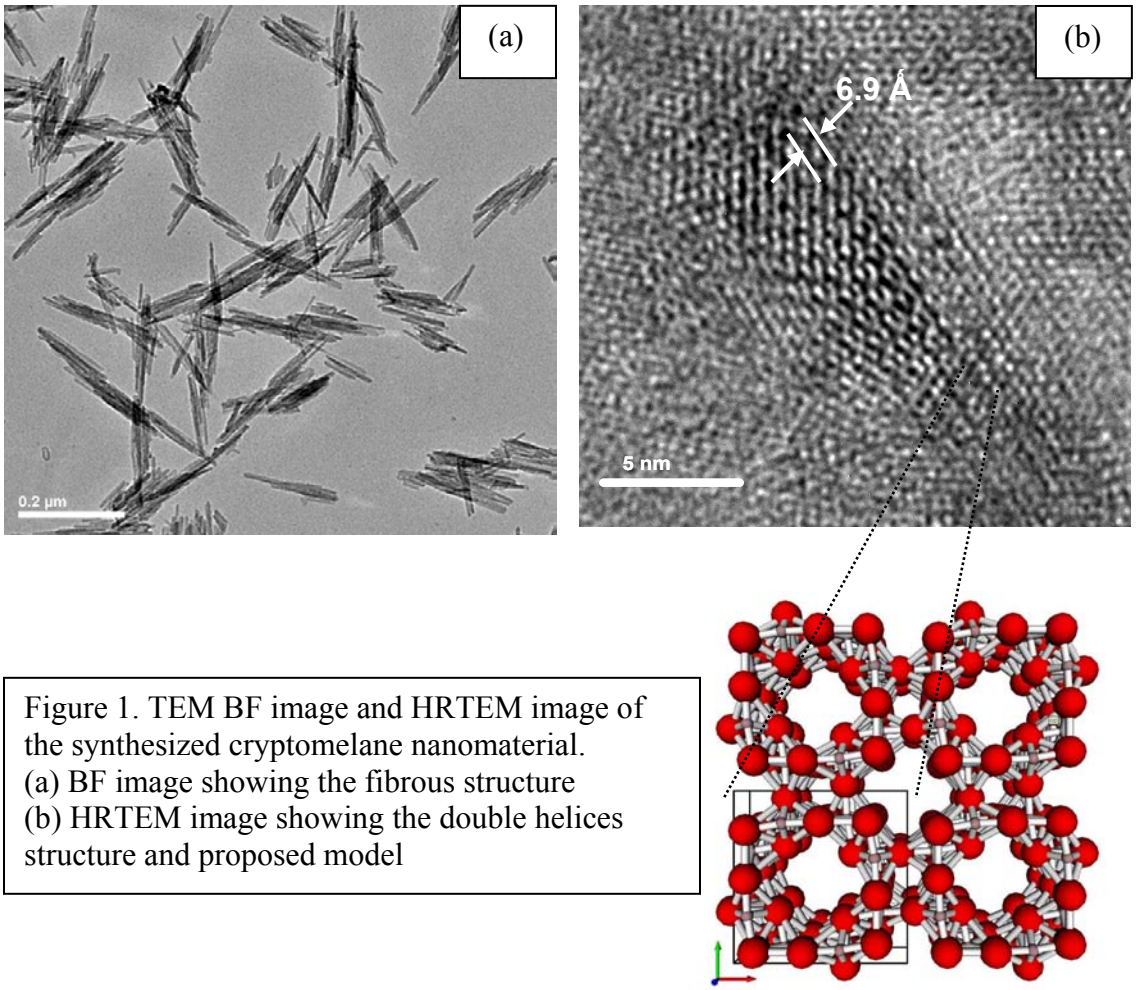

Figure 1. TEM BF image and HRTEM image of the synthesized cryptomelane nanomaterial.
(a) BF image showing the fibrous structure
(b) HRTEM image showing the double helices structure and proposed model

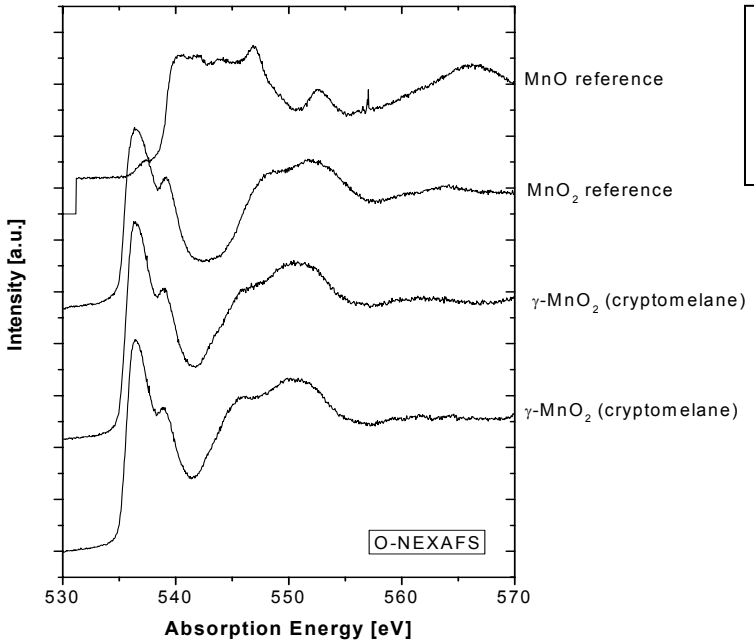

Figure 2. Oxygen K near edge X-ray absorption spectra of the synthesized cryptomelane nanomaterial

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