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NOTE

Growth of Nano (Na)V_2O_5 from $NaBH_4$ and V_2O_5 at Room Temperature

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In this work V_2O_5 was reduced to sodium intercalated NaV_2O_5 with $NaBH_4$ in pyridine-water solvent. Techniques to investigate the composition, crystallinity and morphology of products as prepared include field emission scanning electron microscopy, energy dispersive X-ray spectroscopy and X-ray powder diffraction inductively coupled plasma-atomic emission spectroscopy. Unit cell parameters are a = 11.28, b = 3.583, c = 4.76 Å and $\alpha = \beta = \gamma = 90°$. Mean crystallite size was calculated to be 4.8 nm.

Keywords: Nano (Na)V_2O_5, $NaBH_4$, V_2O_5.

Nanostructured vanadium oxides have attracted special interest because of their wide range of oxidation states (from +2 to +5) and coordination polyhedra in the vanadium-oxygen system[1,2] and special interest because of their outstanding structural flexibility combined with their interesting chemical and physical properties for catalytic and electrochemical applications[3-5]. For example, sodium vanadium oxide bronze (NaV_2O_5) with layered orthorhombic structure has been thought to be one of the most promising cathode materials for lithium-ion batteries due to its low cost, high discharge rate and long cycle life[5-13].

Various methods have been used to fabricate nanostructured vanadium oxide bronze including spark plasma[14], electrochemical insertion[7] and chemical vapor deposition[15]. In this report, NaV_2O_5 was synthesized with reduction of V_2O_5 with $NaBH_4$ in solution phase at room temperature.

All of the reagents were analytical grade without further purification. 0.182 g (1 mmole) V_2O_5 and 0.189 g (5 mmole) $NaBH_4$ were mixed in pyridine-water (1:1) solution for 24 h in open air at room temperature. Dark green precipitate was filtered, washed two times with distilled water and dried. Then, sample is calcined 500 ºC for 5 h.

X-ray powder diffraction measurement were carried out with ARL Xtra X-ray diffractometer device with CuK_α radiation in the 2θ range of 10-80º. Mean crystallite size was calculated with Williamson-Hall method. The morphology of the material was studied with a field emission scanning electron microscopy (FE-SEM, JSM-6700F) performed at 5 kV . The composition of the material was determined by energy dispersive X-ray spectroscopy (EDS, OXFORD INCAX Sight Resol.133ev) and inductively coupled plasma-atomic emission spectroscopy (PE®, ICP-AES/1000).

The phase structure of the resulting product was determined by X-ray powder diffraction (Fig. 1). It is obvious that the crystalline phases for sodium vanadium oxide nanocrystals are discriminatory. Indeed, the diffraction pattern of the obtained product consists of a series of peaks of multiple reflections characteristic of lamellar structures (presence of a peaks which has d-spacing's of the type d001/l, where l is an integer). This series can be perfectly indexed to orthorhombic vanadium oxide crystalline phase V_2O_5 with lattice constants of a = 11.28, b = 3.583, c = 4.76Å Furthermore, the XRD patterns display a 00l set of reflections with high intensity corresponding to the stacking of the layers along a direction perpendicular to the substrate, presenting a well-ordered layered structure. The interlayer distances are indicated from the peaks with the highest intensity at low diffraction angle and characterized by 001 Miller index (d001 = 4.797(6)Å). This observation indicates that the sodium is sandwiched between vanadium oxide layers and organized them into a single direction. The average crystallite size of the as-synthesized materials was calculated by using Willi-amson-Hall method. The average crystallite size values, calculated from XRD patterns of the samples have been found to be 4.8 nm.

The morphology of synthesized samples was investigated by using the field emission scanning electron microscopy (FE-

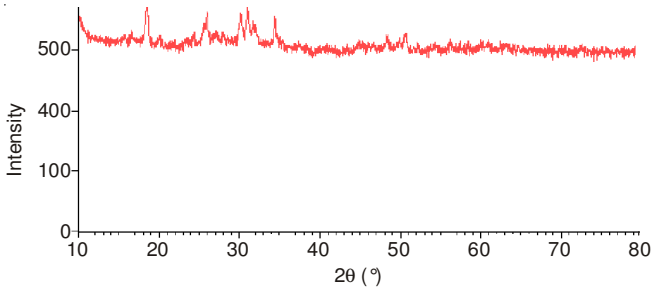

Fig. 1. XRD patterns of NaV_2O_5

SEM). Fig. 2 shows FE-SEM image of the sample synthesized at room temperature for reaction time of one day. Indeed, the SEM image reveals that the as-obtained material is made of the homogenous phase with non uniform particles which display different morphology. In fact, the as synthesized particles are up to several of nanometers to micrometers. Results of ICP-EAS and EDX analysis proved the formation of NaV_2O_5.

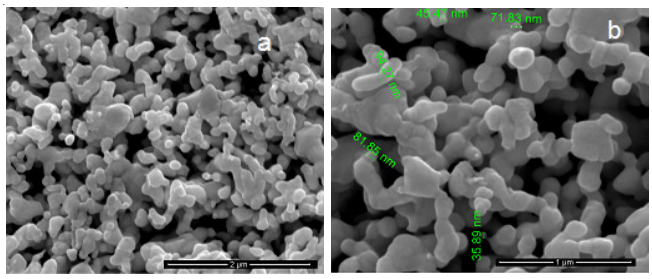

Fig. 2. FE-SEM images of NaV_2O_5

Conclusion

Nano and micro sized sodium intercalated NaV_2O_5 was synthesized successfully in good yields at room temperature.

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