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SYNTHESIS OF α-MnO_2 WIRE LIKE STRUCTURES AND MODIFIED ELECTRODE MATERIAL FOR SUPERCAPACITOR APPLICATIONS

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ABSTRACT

Nanowires a-MnO_2 material has been successfully synthesized via co-precipitation-ultrasonication method. The effect of $KMnO_4$ concentration on the formation of wire like structure of a-MnO_2 has been studied. The obtained material displays the tetragonal phase, which was systematically investigated by powder X-ray diffraction (PXRD) analysis . The formation of wire like structures was confirmed by scanning electron microscopy (SEM). The electrochemical performance of the modified a-MnO_2 electrode material has been analyzed by cyclic voltammetry (CV) and charge-discharge curve (CP) using three electrode system. The modified electrode material shows higher specific capacitance of about 293.51 F/g at 1 A/g current density in 1 M KCL electrolyte solution. The significance of this modified electrode material was to realize the rectangular shaped in the CV curve. The high capacitive performance of a-MnO_2 material is attributed to the one dimensional structure. The electrode reveals the pseudocapacitance behavior, which is attributed to the redox contribution of MnO_2/MnOOH in alkaline medium that makes this material attractive for potential charge storage (pseudocapacitor) applications.

INTRODUCTION

In recent years, the development of alternative energy conversion systems that can meet present day power demands has accelerated due to the environmental issues. Supercapacitors are the energy storage devices having higher energy density than conventional capacitor and a greater power densities and a longer life cycle than those batteries [1]. According to the charge storage mechanism, supercapacitor can be classified into two types: (i) electrical double-layer capacitor (EDLC), which is based on non-faradic charge separation at the electrode/electrolyte interface (carbon based electrode materials are commonly used), and (ii) pseudocapacitor, which is based on faradic redox reactions of electro-active materials (metal oxides and conductive polymers are commonly used) [2]. Transition-metal oxides are an important class of materials for variety applications in electrochemical supercapacitors. Among the various metal oxides, Manganese dioxide (MDO) has attracted great attentions as supercapacitor electrode materials since 1990's, Owing to intriguing characteristics of easy preparation, low cost, environmental friendliness, excellent electrochemical properties and safe material. A large theoretical specific capacitance (1370 F/g), suggesting that it as the most promising electrode material for supercapacitors. MnO_2 possesses the different crystallographic forms, such as α-β-γ-δ and ε-type, where the main structural α-MnO_2 has nanoscale 2 x 2 tunnel structure made of MnO_6 octahedral sharing edges and corners with different connectivity [3]. Among all these manganese dioxide, α-MnO_2 attracts great interest, due to its good electrochemical capacitance behaviors. The substantial efforts have been made towards the development of nanostructured materials for electrochemical energy storage, which offer enhanced electrochemical performance because of the ability to access both bulk and surface properties. The advantages in using nanostructured materials due to fast charge-discharge rates and excellent cycling stability have been reported. These remarkable results are generally attributed to the short path lengths for both electronic transport as well as the large active surface area of the nanomaterials [4]. In fact, the electrochemical properties of the MnO_2 strongly depend on its particles size, morphology and crystalline structure. Furthermore, the several techniques have been developed for the preparation of nanostructured MDOs with significant progress [5]. Nanosized MDOs for electrode materials are typically fabricated via electrochemical or chemical routes. Recently, Q. Li et.al, studied the electrochemical properties

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of the MnO_2 nanowires [6] and the Nanostructured MnO_2 material with nanorods was investigated by Y. Liu et.al [7]. In this present work, we have synthesized template free α-MnO_2 wire like structure by the wet-chemical method. The structural and morphological properties of material were investigated by powder X-ray diffraction (PXRD) and Scanning electron microscopy (SEM). We would like to address the important of the electrochemical properties of the modified electrode for supercapacitor applications.

EXPERIMENTAL

For α-MnO_2 was synthesized by oxidation of manganese acetate by potassium permanganate in an aqueous solution. The stoichiometric amount of $Mn(II)(CH_3COO)_2$ and $KMnO_4$ was dissolved in 100 mL ethanol and stirring was continued for 5 h at 60°C. A black precipitate was formed. The precipitate was separated by centrifugation after 5 times ultrasonication process for 15 minutes. Subsequently, the product was washed with several times of ethanol and water for further purification. The obtained product was dried at 100°C for overnight in air oven. Then, the final product was subjected to heat treatment at 450°C for 5 h in an ambient atmosphere.

RESULTS AND DISCUSSION

Powder XRD Analysis

The crystallinity and phase of the product was examined by powder X-ray diffraction as shown in Fig.1. The XRD shows cryptomelane-like MnO_2 phase. The peaks are indexed to tetragonal phase with I4/m space group according to the [JCPDS 44-0141] standard file. The broad peak at 37.1° indicates the formation of the α-MnO_2. No extra peaks related to impurities were observed and demonstrate the high purity of the final product. In general, the MnO_2 exhibits the different crystal phases such as, α,β,γ,δ,λ and ε. These phases are distinctive from each other due to the basic unit of [MnO_6] octahedral are linked in different ways. Consequently, the different phases possess tunnel or interlayers with gaps of different magnitude. The a-MnO_2 phase constructed from double chains of [MnO_6] octahedral, which are linked at the corners to form a 2×2, 1×1 tunnel structure extend to the direction of c axis of tetragonal unit system. For the reason for using α-MnO_2, the 1x1 tunnel size has empty because of their size 1.49 nm and the size of the 2×2 about 0.46 nm, which is suitable for interaction/extraction of alkaline cations [8].

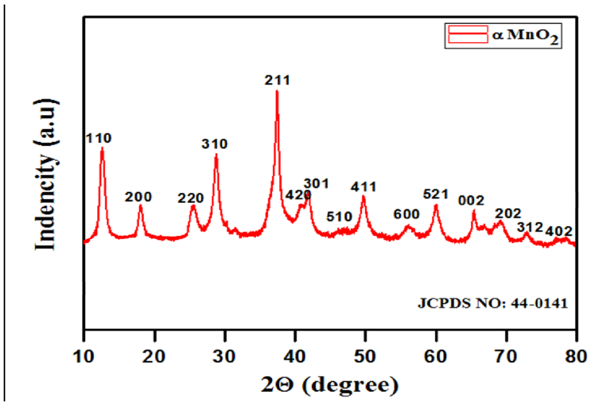

Fig. 1. The typical XRD pattern of MnO_2 prepared material.

Fig.2. Shows the surface morphology of the a-MnO_2 product. The higher magnification SEM image illustrates wires like structure. For the active material, nanostructures have larger specific surface area and shorter diffusion distance, which can provide faster probability, higher material utilization, and better rate performance [9].

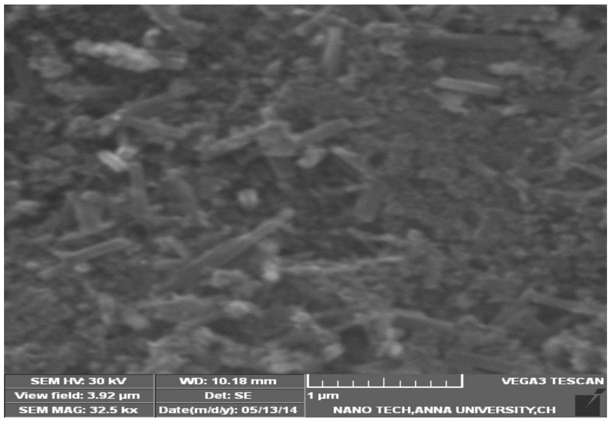

Fig. 2. The SEM image of MnO_2 prepared material.

Cyclic Voltammetric Analysis (CV)

The electrochemical properties of the modified electrode have been investigated by using CV tests between the potential ranges of -0.1 to 0.6 V vs. Ag/AgCl. The CV curves recorded at different sweep rates in 1M KCl electrolyte are shown in Fig. 3. The two mechanisms are proposed for the charge storage in MnO_2 based electrodes i.e. (i) the first based on the concept of intercalation of H^+ or alkali metal cations such as K^+ in the electrode during reduction and de-intercalation upon oxidation,

$$MnO_2 + H^+ + e^- \leftrightarrow MnOOH \quad (1)$$

(or)

$$MnO_2 + C^+ + e^- \leftrightarrow MnOOC$$

$$(MnO_2)_{surface} + C^+ + e^- \leftrightarrow MnO_2\,(C^+)_{surface} \quad (2)$$

(ii) The second based on the surface adsorption of electrolyte cations (C^+) on MnO_2. Where, $C^+ = Na^+, K^+, Li^+$ [10-11].

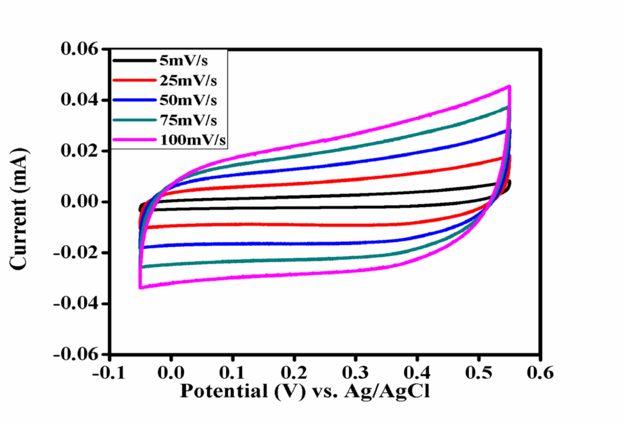

Fig. 3. The cyclic volttametry curves of modified electrode material at different scan rates.

The CV curves at different scan rates show no redox and oxidation peaks, indicating that the electrode is charged and discharged at a pseudoconstant rate over the complete voltammetric cycle. While the CV curve shows the nearly ideal rectangular shape with mirror image feature at low scan rate, which means that manganese oxide electrodes have better reversibility and ideal capacitive property when discharging with lower current density [12]. The synthesized a-MnO_2 indicates the high specific capacitance behavior, which attributes to their crystal structure and morphology. First, the crystal structure of prepared material of α-MnO_2 was constructed from double chains of octahedral [MnO_6] which is from the 2×2 tunnels. Secondly, the nanowires microstructure possesses the large electrode/electrolyte contact area and surface adsorption and desorption of alkaline cations [8]. It shows almost ideal capacitive behavior. It is well known that a high capacity material like α-MnO_2 is a potential electrode for supercapacitor Applications

Charge–discharge study (CP)

The charge–discharge study of the modified electrode was investigated from -0.1 to 0.6 V at various current densities in 1M KCl electrolyte as shown in Fig. 4. The obtained charge and discharge curves were show the linear and symmetrical behavior.

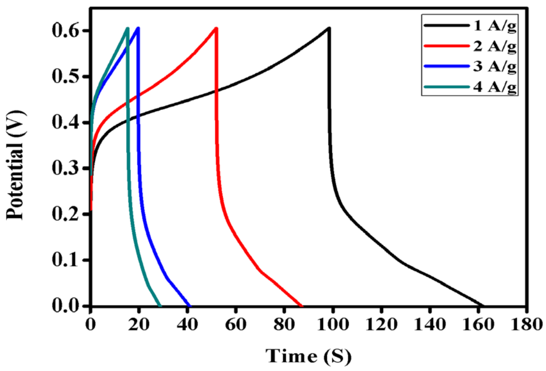

Fig. 4. The charge-discharge curves of modified electrode material at different scan rates.

It implies that the electrochemical cells have excellent electrochemical reversibility and capacitive properties [13]. The specific capacitance of the electrode was obtained using from the following equation,

$$Cs = (I \times \Delta t)/(\Delta V \times m)$$

Where m is the mass load on the active-electrode material, ΔV is the potential window, I is the dicharge current, Δt is the total discharge time, and Cs is the specific capacitance. In this way, we obtained the maximum specific capacitance of the prepared material was 293.51 F/g in 1 A/g current density. The charge -discharge characteristics are good agreement with the result obtained from CV curves.

CONCLUSION

In this present, we had successfully synthesized Manganese dioxide (MDO) system without any secondary phase formation as confirmed by XRD. SEM image show the wires like structures making it easier ion diffusion. This nanostructured electrode material plays an important role in redox process. The CV curves show an ideal capacitive behavior with high capacity and identified as one of the potential electrode material for supercapacitor applications.

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