

FIRST INTERNATIONAL CONFERENCE ON ELECTRON MICROSCOPY OF NANOSTRUCTURES

ELMINA  2018

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Synthesis and Characterization of MnCo_2O_4 Porous Spinel Oxide

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This work presents an investigation on spinel structured material that consist of Mn(II) and Co(II) combined in the formula MnCo_2O_4 , where Mn(II) occupies tetrahedral and Co(II) octahedral sites of crystal structure. Such spinel structured material, MnCo_2O_4 was synthesized by citrate-gel combustion (CGC) technique, carefully chosen as the method of synthesis is very important for producing a material with desirable physico-chemical characteristics. The CGC method of synthesis is well known for production of nanodispersed simple or complex oxides, catalyst and superconductors [1]. This sol-gel auto combustion technique is quite common, provides a very good homogeneity of samples, very easy control of stoichiometry and delivers production at low cost. The proposed method involved nitrate salt as oxidizer and citric acid as a reducing agent (fuel). The molar ratio of nitrate salt precursors was set to 0.5:0.5. The choice of fuel, as well as the ratio of oxidizer/fuel affects morphology of spinel porous material [2]. The molar ratio of citric acid versus nitrate groups was 1:3.6. In preparation of MnCo_2O_4 , aqueous ammonia (1 M) was added into mixture of $\text{Mn}(\text{NO}_3)_2$, $\text{Co}(\text{NO}_3)_2$ and citric acid aqueous solution (in order to adjust the to pH=7), subsequently followed by water evaporation and heating under constant stirring until a light pink sol was formed. The sol turned into gel and this was finally calcinated for 2h at 450 °C. The equation of combustion of citric acid is $2\text{C}_6\text{H}_8\text{O}_7 + 9\text{O}_2 = 12\text{CO}_2 + 8\text{H}_2\text{O}$. Since the temperature needed to complete combustion of remaining carbon residues is unknown, the heating at a constant temperature of 500° C was prolonged for approximately 30 minutes, which was enough to obtain carbon-free oxides. After calcination, a black powder of MnCo_2O_4 nanoparticles was obtained. The physico-chemical characterization of as-prepared

material was performed by means of X-ray diffraction (XRD) and scanning electron microscopy (SEM). A representative X-ray diffraction pattern of the as-prepared final product is shown in Fig. 1. Obtained X-ray spectrum shows to be in good agreement with standard pattern of spinel structure of MnCo_2O_4 (JCPDS card no. 23-1237) regarding some of the diffraction peak positions. The most intense lines of 2θ at 18.53° , 30.47° , 35.99° , 43.64° , 54.30° , 57.84° , 63.45° in XRD spectrum are in good agreement with the angles 18.55° , 30.54° , 35.995° , 43.759° , 54.336° , 57.909° and 63.622° in JCPDS card of MnCo_2O_4 . As-prepared MnCo_2O_4 shows some noisier XRD spectrum than standard pattern of spinel structure of MnCo_2O_4 and there are no impurity peaks. These noises are due to the low crystallite size of the prepared material. Debye-Scherrer formula was used to calculate the average crystallite size of the prepared MnCo_2O_4 which around 19 nm [3,4]. SEM image (Fig. 2) shows porous morphology of MnCo_2O_4 material. Homogeneity of oxide mixtures was also visible which means that a fine dispersion of Mn and Co oxides prevailed, probably achieved due to the equal molar ratio of Mn and Co oxides in the mixture. Elemental composition analysis of this porous spinel material obtained from energy dispersive spectroscopy (EDS) further confirms the existence of Mn, Co and O with calculated composition of 2.21 wt%, 2.99 wt% and 11.33wt%, respectively (Fig. 3). The synthesis of this porous material proved to be inexpensive, fast and environmentally friendly and the material could be potentially used as an electrochemical sensor, especially in the field of heavy metal detection.

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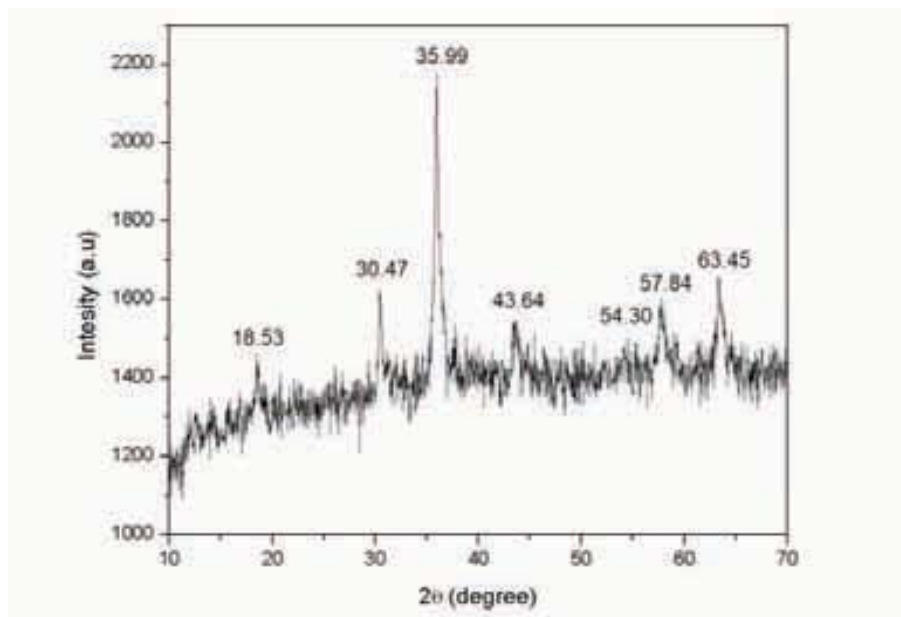


Figure 1. XRD pattern of MnCo_2O_4 spinel oxide.

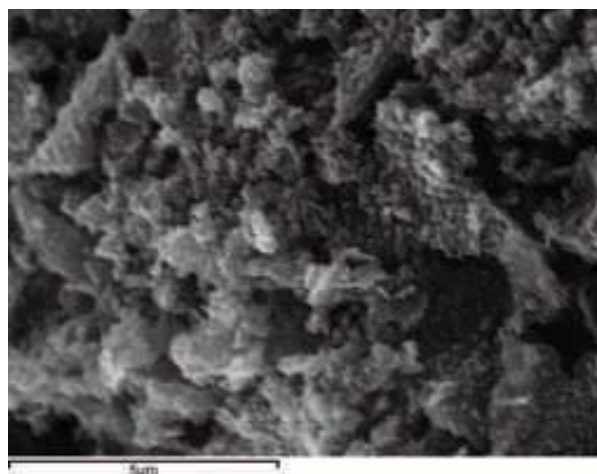


Figure 2. SEM image of prepared porous MnCo_2O_4 .

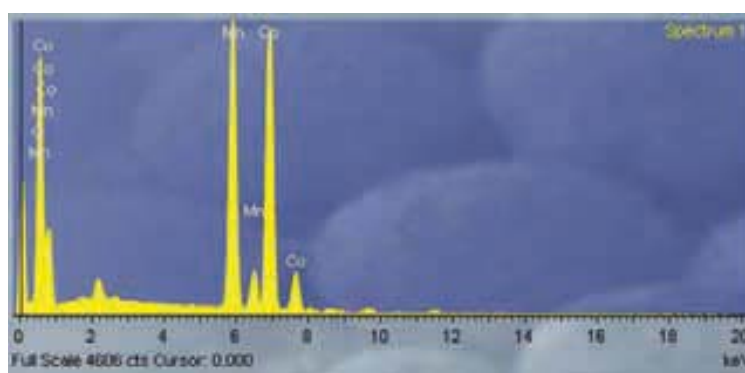


Figure 3. EDS spectrum of prepared MnCo_2O_4 .