

ORIGINAL RESEARCH PAPER

Synthesis, Characterization and Investigation Magnetic and Photovoltaic Properties of FeVO₄ Nanoparticles

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ARTICLE INFO.

Received 05/11/2015

Accepted 10/12/2015

Published online 01/01/2016

KEYWORDS

FeVO₄

Nanoparticle

Solar cell

Sonochemical method

ABSTRACT

This research reports a facile ultrasonic approach for the synthesis of iron vanadate (FeVO₄) nanoparticles with the aid of ammonium metavanadate (NH₄VO₃) and Fe(NO₃)₃·9H₂O as the starting reagents without adding external surfactant, capping agent or template in an aqueous solution. Furthermore, to examine the solar cell application of as-synthesized iron vanadate (FeVO₄) nanoparticles, FTO/TiO₂/FeVO₄/Pt-FTO structure was created by deposited iron vanadate film on top of the TiO₂ layer. The VSM magnetic measurement result shows that a maximum magnetization of 0.16 emu/g was obtained for FeVO₄ nanoparticles after annealing at 550 °C for 150 min. The as-synthesized FeVO₄ ferrite powders were characterized by X-ray diffraction, scanning Electron Microscopy, spectra energy dispersive analysis of X-ray, and vibrating sample magnetometer techniques. The X-ray diffraction study showed that pure anorthic phase of FeVO₄ nanoparticles have been produced after calcinations at 550 °C for 150 min. Solar cell result indicates that an inexpensive solar cell could be developed by synthesis of FeVO₄ nanoparticles through the ultrasonic approach.

INTRODUCTION

In the past few decades, the studies of nanoparticles system have been extensively a subject of continue interest in chemistry, physic, and other fields [1-5]. Multiferroics are new class of materials, attracting attention because of simultaneous presence of more than one ferroic orders, especially magnetic and ferroelectric ordering [6-7]. Transition metal based orthovanadates (AVO₄) are the cohesive class of materials which have potential applications in various fields. So far, different vanadates such as BiVO₄, CeVO₄, LaVO₄, PrVO₄, GdVO₄, and NdVO₄ [8-13], have been synthesized and characterized. They have been used as catalysts, cathode materials in lithium ion batteries, gas sensors, solar cells, photoluminescence, microwave applications, optical fibres, scintillator

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materials, and humidity sensors. Among the metal vanadates, FeVO₄ is a highly stable and highly selective catalyst that finds many applications including photo catalytic degradation of the organic pollutants [14], gas sensor materials in detecting H₂S trace in air environment [15], electro chromic electrodes [16] and various other applications. FeVO₄ has four different polymorphs named as FeVO₄-I, FeVO₄-II, FeVO₄-III, and FeVO₄-IV, of which FeVO₄-I is a stable phase formed at room temperature while others are metastable phase that are usually formed at high pressure and high temperature [17]. In FeVO₄-I, Fe³⁺ ions have three crystallographic sites of which two sites are distorted octahedral FeO₆ and one site is in distorted trigonalbipyramidal FeO₅ environment. Fe-O polyhedra forms 6 columns doubly bent chain and the chains are joined together by VO₄ tetrahedra to form three dimensional frame works [18]. Among various

methods for the preparation of nanostructures, ultrasonic method is more promising in terms of low cost, simply control the shape, and particle size, low processing temperature, simplicity and potential for large-scale production. Recently, the ultrasonic process as a fast, convenient, and economical method has been widely used to generate novel nanostructure materials under ambient conditions [19-22]. Therefore, in this study in order to extend the ultrasonic method for preparation of inorganic vanadate nanoparticles, we report the preparation of FeVO_4 nanoparticles by ultrasonic method in an aqueous solution. The as-synthesized FeVO_4 nanoparticle was utilized as the photo-anode material for fabrication of $\text{FTO}/\text{TiO}_2/\text{FeVO}_4/\text{Pt-FTO}$ solar cell structure to examine its solar cell application. Furthermore, the nature of magnetization applied field (M-H) infers paramagnetic behaviour for FeVO_4 nanoparticles.

MATERIALS AND METHODS

Characterization

X-ray diffraction (XRD) pattern was recorded by a Philips-X'PertPro, X-ray diffractometer using Ni-filtered $\text{Cu K}\alpha$ radiation at scan range of $10 < 2\theta < 80$. Scanning electron microscopy (SEM) images were obtained on LEO-1455VP equipped with an energy dispersive X-ray spectroscopy. The energy dispersive spectrometry (EDS) analysis was studied by XL30, Philips microscope. Photocurrent density-voltage (J-V) curve was measured by using computerized digital multimeters (Ivium-n-Stat Multichannel potentiostat) and a variable load. A 300 W metal xenon lamp (Luzchem) served as assimilated sun light source, and its light intensity (or radiant power) was adjusted to simulated AM 1.5 radiation at $100 \text{ mW}/\text{cm}^2$ with a filter. The magnetic measurement of sample was carried out in a vibrating sample magnetometer (VSM) (Meghnatis Daghigh Kavir Co.; Kashan Kavir; Iran) at room temperature.

Fabrication of $\text{FTO}/\text{TiO}_2/\text{FeVO}_4/\text{Pt-FTO}$ Cell

Electrophoresis deposition (EPD) was utilized to the prepare TiO_2 films. During EPD, the cleaned FTO glass remained at a positive potential (anode) while a pure steel mesh was used as the counter (cathode) electrode. The linear distance between the two electrodes was about 3 cm. Power was supplied by a Megatek Programmable DC Power Supply (MP-3005D). The applied

voltage was 10 V. The deposition cycle was repeated 4 times, each time 5s, and the temperature of the electrolyte solution was kept constant at 25°C . The coated substrates were air dried. The apparent area of the film was $1 \times 1 \text{ cm}^2$. The resulted layer was annealed under an air flow at 500°C for 30 min. Electrolyte solution consisted of 120 mg/l of I_2 , 48 ml/l of acetone, and 20 ml/l of water. For deposition of FeVO_4 powder on the FTO glass substrate, a paste of FeVO_4 was initially prepared. The slurry was produced by mixing and grinding 1.0 g of the nanometer sized FeVO_4 with ethanol and water in several steps. Afterwards, the ground slurry was sonicated with ultra-sonic horn (Sonicator 3000; Bandeline, MS 72, Germany) and then mixed with terpeneol and ethyl cellulose as binders. After removing the ethanol and water with a rotary-evaporator, the final paste was prepared. The prepared FeVO_4 paste was coated on TiO_2 film by a doctor blade technique. After that the electrode was gradually heated under an air flow at 450°C for 30 min. Counter-electrode was made from deposition of a Pt solution on FTO glass. Afterwards, this electrode was placed over $\text{TiO}_2/\text{FeVO}_4$ electrode. Sealing was accomplished by pressing the two electrodes together on a double hot-plate at a temperature of about 110°C . The redox electrolyte consisting of 0.05 M of LiI, 0.05 M of I_2 , and 0.5 M of 4-tert-butylpyridine in acetonitrile as a solvent was introduced into the cell through one of the two small holes drilled in the counter electrode. Finally, these two holes were sealed by a small square of sealing sheet and characterized by I-V test.

Synthesis of FeVO_4 nanoparticles

All the chemicals used in this method were of analytical grade and used as-received without any further purification. Ultrasonic irradiation was accomplished using a high-intensity ultrasonic probe (Sonicator 3000; Bandeline, MS 72, Germany, Tihorn, 20 kHz , 60 W cm^{-2}) immersed directly in the reaction solution. At first, 1 mmol of NH_4VO_3 was dissolved in 15 ml of distilled water under ultrasonic irradiation at room temperature. In the second step, 1 mmol of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ was dissolved in 15 ml of distilled water and was added drop-wise to the above solution under stirring. Then, the final mixtures were loaded into a beaker where the reaction was performed in an ultrasonic digestion system at 75 w for 10 min. Afterwards, the product was dried in vacuum at 80°C for 3 h. Finally, the as-prepared products by ultrasonic

method were calcined at 550 °C in air for 2.5 h.

RESULTS AND DISCUSSION

The XRD pattern of the as-synthesized FeVO₄ nanoparticles at 75 W for 10 min is shown in Fig. 1. Extremely broaden reflection peaks were observed in Fig. 1, which indicated fine particle nature of the obtained anorthic phase of FeVO₄ nanoparticles (space group P-1, JCPDS No. 71-1592) with the calculated cell parameter of a= 6.7190 Å, b= 8.0600 Å, and c= 9.2540 Å. No other crystalline phases were detected in the calcined product. From XRD data, the crystallite diameter (D_c) of FeVO₄ nanoparticles (75 W for 10 min) was calculated to be 25 nm using the Scherer equation [23]:

$$D_c = K\lambda/\beta\cos\theta \tag{1}$$

where β is the breadth of the observed diffraction line at its half intensity maximum, K is the so-called shape factor, which usually takes a value of about 0.9, and λ is the wavelength of X-ray source used in XRD.

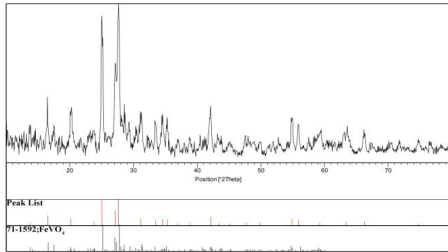


Fig. 1. XRD pattern of FeVO₄ nanoparticles

EDS analysis was used to evaluate the chemical composition and purity of final products (Fig. 2). The EDS spectrum of FeVO₄ nanoparticles shows the presence of Fe, O, and V elements in these nanoparticles. Furthermore, neither N nor C signals are

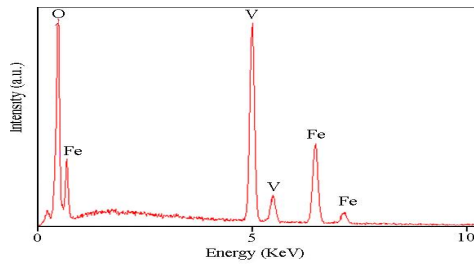


Fig. 2. EDX pattern of FeVO₄ nanoparticles

detected in the EDS spectrum, which means the product is pure and free of any surfactant or impurity.

The morphology of the FeVO₄ nanoparticles has been examined by SEM image (Fig. 3). According to the Fig. 3, it is seen that the products composed of small spherical shapes nanoparticles with average size of about 35 nm.

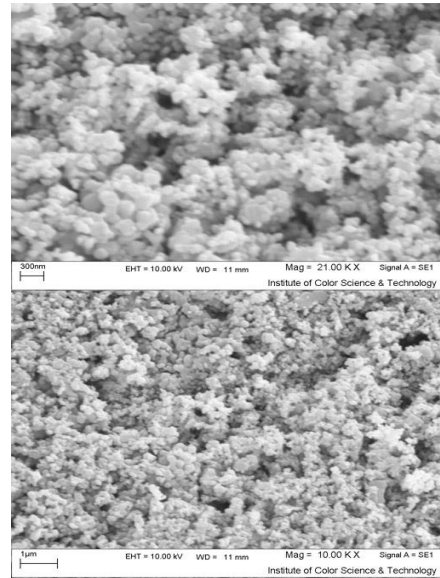


Fig. 3. SEM image of FeVO₄ nanoparticles.

The magnetization (M) of the FeVO₄ nanoparticles as a function of external applied magnetic field (H) at room temperature was measured. Fig. 4 shows the hysteresis loop of FeVO₄ nanoparticles. M-H curve exhibit magnetization of 0.17 emu/g and is an indicative of paramagnetic behavior.

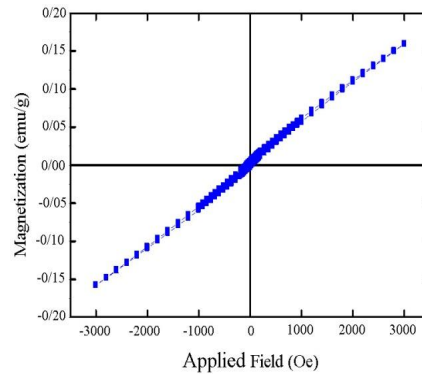


Fig. 4. VSM curve of FeVO₄ nanoparticles

I-V characterization of a typical solar cell fabricated using in situ approach is shown in Fig. 5. The measurement of the current density voltage (I-V) curve for FeVO₄ nanoparticles was carried out under the illumination of AM1.5 (100 mW/cm²). Device characteristics are as follows: V_{oc} = 0.3 V, J_{sc} = 0.133 mA/cm², η = 0.13, and FF = 3.33 for FTO/TiO₂/FeVO₄/Pt-FTO.

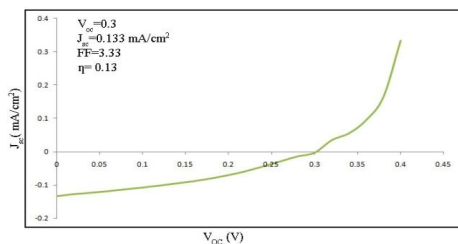


Fig. 5. I-V characterization of the FTO/TiO₂/FeVO₄/Pt-FTO.

CONCLUSIONS

In summary, we have demonstrated the synthesis of FeVO₄ nanoparticles from ammonium metavanadate powder by ultrasonic process. The nature of M-H curve represents paramagnetic behaviour in FeVO₄ nanoparticles. A preliminary study on the possibility of developing a solar cell having FTO/TiO₂/FeVO₄/Pt-FTO structure was also performed.

ACKNOWLEDGMENT

Authors are grateful to University of Arak and VSM institute, Meghnatis Daghigh Kavir Co.; Kashan Kavir; Iran, for providing financial support to undertake this work.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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How to cite this article:

Sobhani-Nasab A, Hosseinpour-Mashkani S.M, Maddahfar M. Synthesis, Characterization and Investigation Magnetic and Photovoltaic Properties of FeVO₄ Nanoparticles. J. Nanostruct. 2016; 6(1): 70-73.

DOI: 10.7508/jns.2016.01.011

URL: http://jns.kashanu.ac.ir/article_13647.html