RESEARCH ARTICLE

Evaluation of heat temperatures on the structure of nano-rod

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ABSTRACT

In this project, the TiO_2 nano-rods were synthesized from P25 TiO_2 nanoparticles by hydrothermal method in 20 M NaOH solution. The effects of annealing temperatures on produced nanorods were investigated by scanning electron microscopy (SEM) X-ray diffraction (XRD) and photoluminescence (PL) spectroscopy. Increase of annealing temperature over 300 °C has no effect on the size of produced nanorods, but due to reducing of the surface charge causes the more compact products. In addition, the surface defects of synthesized 1D TiO_2 were decreased because of increase of annealing temperatures, but we should note that at higher tempera tures (over 500 °C) the TiO_2 phase may be converted from anatase to rutile.Diameter growth and changes of surface oxygen defects of synthesized Nano rods are studied with increasing annealing temperatures. Also, with Increase of annealing temperature over 300 °C there is no effect on the size of produced nano-rods, but due to reducing of the surface charge causes the more compact products.

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INTRODUCTION

Nowadays, the nanostructured metal oxide semiconductors widely employed due to their promising applications in electronic, optical, chemical and mechanical properties [1-2]. Among the various metal oxide semiconductors, titanium dioxide (TiO2) has attracted great attention because of its potential to apply in environmental cleaning, photo-catalysis, sensors, lithium ion batteries and solar cells [3]. Therefore, many researches recently have been done to improve and recognize the structures and properties of TiO2nanostructures. One of the most attractive research areas was synthesized and investigated one dimensional (1D) TiO2 nanostructures, because of their potential applications in a variety of novel devices [4]. Various TiO2-derived 1D nanostructures (nanotube, nanorod, nanowire, nano-fiber) have been synthesized by various techniques, such as template method, anodic oxidation, and hydrothermal method [5]. The last technique was introduced in 2015 by Kasuga et al. [6] there are much research has been targeted towards an improved understanding of the mechanism of 1D TiO2 formation in alkali solution

to establish the crystal structure of the nanostructure [7]. Titania 1D nanostructures could be synthesized by a reaction of NaOH and TiO2 nanoparticles with anatase or rutile structure. It has been generally recognized that the obtained nano-structures are built in titanate compounds such as M2Ti3O7 or MxH2- xTi3O7 (M = Na or K) [8]. Normally raw TiO2 nanoparticles, such as a commercial anatase powder

P25 (Degussa Co., Germany, a mixture of anatase and rutile; its size is less than 30 nm), are usually used as the raw powder reacted with NaOH or KOH [9]. In this paper, we have investigated the effect of post heat treatment on microstructure and morphology of synthesized TiO2 nanorod by hydrothermal method. We have also presented detailed scanning electron microscopy (SEM) studies on morphology and structures of titanate nanorod. In addition, the effect of post heat treatment on surface oxygen vacancies and defects

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of producing TiO2 nanorod has been investigated by photoluminescence (PL) spectroscopy [10].

EXPERIMENTAL

The TiO2 source used for producing the nanorod was a commercially TiO2 powder (P25, Degussa AG, Germany), which consists of 20% rutile and 80% anatase. The primary particle size was about 21 nm. For preparing TiO2 nanorod 1.5 g of TiO2 powder was added to 100 ml of 10M NaOH solution. Next, the TiO2 powder was dispersed by ultrasonic bath (15 min) and vigorous stirring (30min) respectively. The solution was placed in a PTFE (Teflon) lined autoclave and heated for 48 hours at 150 °C. After the hydrothermal treatment, the resulting powder was cooled to room temperature, and then the filtered precipitate washed with water and 0.1M HCl solution until the washing solution achieved pH 6 ~ 7.At the end, the washed powder was dried in oven at 100 °C for 24 hours. The obtained powder was sintered at 300 and 500 °C to study the post heat treatment effects on the TiO2 nanorods morphology and structure. The morphologies of prepared TiO2 nanorods were characterized by scanning electron microscopy (SEM, Hitachi S-4160). The crystal structure was analyzed from XRD patterns (using $Cu - Ka$ radiation, BRUKER, D8 advance). The adsorption/desorption isotherms of these samples were measured by N2-adsorption at 77 K (BEL, Japan Inc) using Barrett-Joyner-Halenda (BJH) and Brunauer-Emmett-Teller (BET) calculations to characterize the porosity and surface area of the prepared nanorod, respectively. In addition, the post heat treatment effects on the synthesized nanorods were characterized by photoluminescence spectroscopy (Jasco, FP 6500).

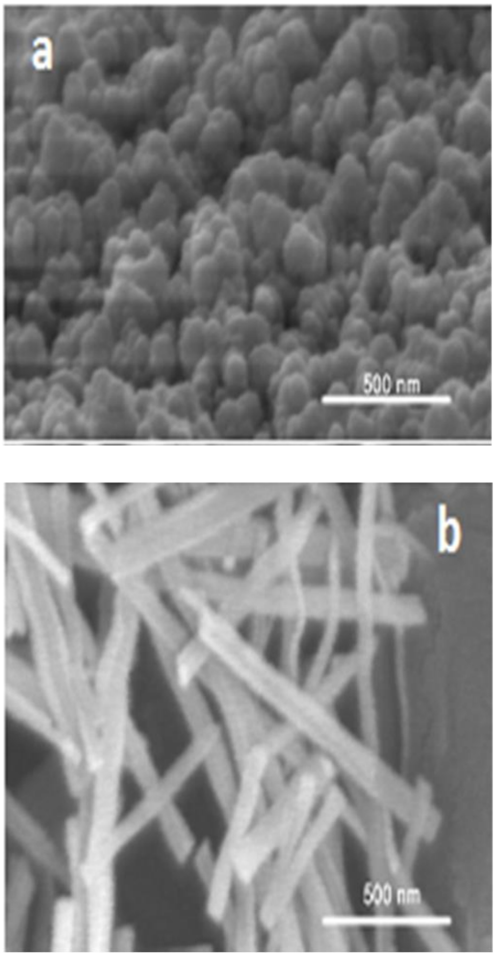

Fig. 1. SEM images of (a) P25 nanoparticles as precursor (b) the synthesized nanorod after 100 °C.

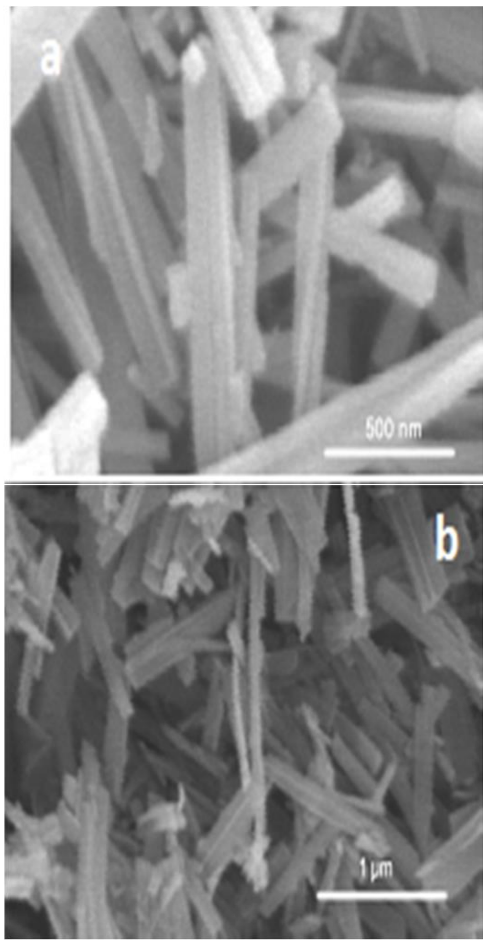

Fig. 2. The SEM images of synthesized nano-rods after a) 300 b) 500 °C.

RESULTS AND DISCUSSION

In Fig. 1.a shows the morphological images of TiO2 nanoparticles as precursor for the hydrothermal nanorod formation. After hydrothermal reaction for 48 h and the post hear treatment for 24 h, TiO2 nanoparticles converted to 1D structure nanorod with about 75 nm and few hundred nm in length (Fig. 1.b). The BET specific surface area (*S*BET), pore volume (*VP*), and pore diameter for prepared nanorod (Fig. 1b) was about 60.06 (m^2/g), 0.27 (cm^3/g) and 18.54 nm, respectively. Adsorption/desorption isotherms of synthesized sample also indicated that TiO2-derived titanate nanorod have mesoporous structures. Fig. 2 shows the SEM images of prepared nanorod after annealing at 300 (2a) and 500 °C. The SEM results indicated that nonorods diameters were increased in results of increasing the post heat treatment at 300 °C, but we didn't observe change in diameter at annealing at 500 °C. The nanorods diameters were become about 125 nm after post heat treatment at temperatures over 300 °C. This may be due to decreasing of surface charge and nanorods approaching together. Another point, the nanorods were more compact with relation to increase of annealing temperatures [11]. As shown in Fig. 3, the X-ray diffraction (XRD) pattern of precursor nanoparticles (P25) is compared with produced nanorods after annealing at 100, 300 and 500 ˚C. As expected, the peaks in the diffraction patterns for the nanorods broadened due to dimensional confinement and the new peaks were become evident in 2θ=28, 46 and 58, because of 1D TiO2 nanostructures[12] . The rutile structures peaks of TiO2 nanoparticles were eliminated after hydrothermal processes and all synthesized sample have an anatase structures (2θ=25, 29, 48 and 63). We didn't anneal the prepared nanorods over 500 ˚C due to possibility of phase conversion from anatase to rutile. The anatase phase of TiO2 structures is more important than rutile for using in photocalytic and solar system because rutile is the most stable phase while anatase possesses superior optoelectronic and photocatalytic properties [13]. We used the synthesized 1D anatase nanorods in dye-sensitized solar cell system and the obtained data will be present elsewhere. We also investigated the effect of post heat treatment on the surface structures by photoluminescence spectroscopy (PL). Fig. 4. shows the PL spectra of synthesized nanorods annealed at different temperatures with excitation wavelength at 300 nm to understand the changes of the nanorods surfaces structures. The excitonic PL intensity of nanorods samples decreases as the calcination temperature increases, which is due to the increase in size of nanorods with increasing calcination temperature. And, this possibly demonstrates that the excitonic PL mainly

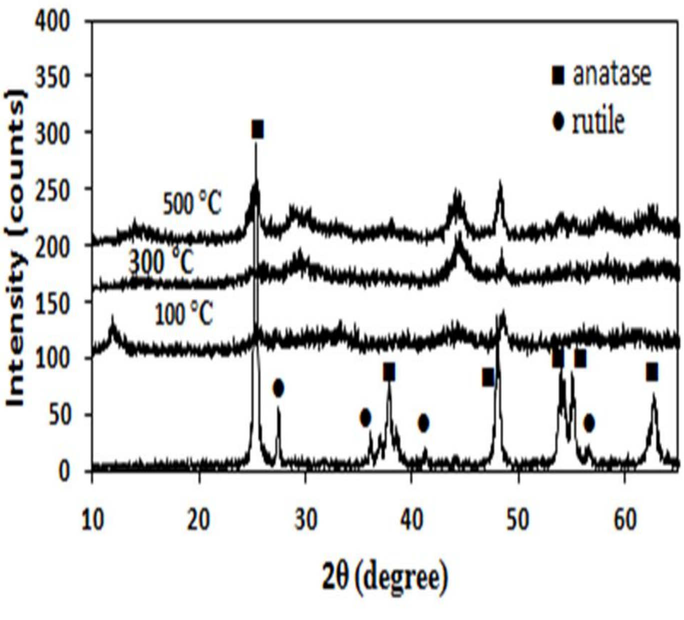

Fig. 3. XRD patterns of prepared nano-rods at different annealing temperatures comparing to P25 TiO2 nanoparticles.

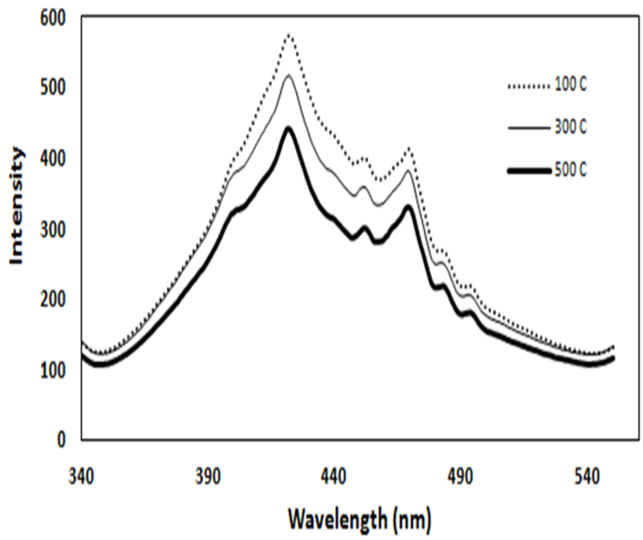

Fig. 4. The PL spectra of synthesized nano-rods at different temperature with excitation wavelength of 300 nm

results from surface oxygen vacancies and defects [15]. Therefore, increasing oxidation of surface of TiO2 with relation to enhance of heat treatment temperatures cause decrease of the surface oxygen vacancies and defects and overall a decrease of PL intensity. On the other hand, reducing the surface defects on TiO2 nanostructures improve the photocatalytic and optoelectronic properties.

CONCLUSION

In summation, the effects of post heat treatment temperatures were in detail investigated by SEM, XRD and PL spectroscopy. The obtained results were shown that the increasing annealing temperature until 300 °C causes increase of thicknesses of prepared nanorods by a hydrothermal method. An increase of annealing temperature over 300 °C has no effect on the size of producing nanorods, but due to reducing of the surface charge causes the more compact products. In addition, the surface defects of synthesized 1D TiO2 were decreased because of increase of annealing temperatures, but we should note that at higher temperatures (over 500 °C) the TiO2 phase may be converted from anatase to rutile.

CONFLICT OF INTEREST

All authors declare that no conflicts of interest exist for the publication of this manuscript.

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