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The Characteristics Study of Titanium Dioxide Nanofibers Prepared by Electrospinning Method

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Abstract

Titanium dioxide nanofibers with diameter ranging to several nanometers were synthesized via electrospinning technique. The precursor solution was prepared by mixing the polyvinylpyrrolidone, PVP (MW∼1,300,000) in ethanol, meanwhile titanium tetraisopropoxide, TTIP in acetic acid was slowly added into the solution under a vigorous stirring. The precursor solutions were then used in the electrospinning process under high voltage supply. As-spun nanofibers were heat-treated under different temperature $400°C$, $500°C$ *and* $600°C$. *The* TiO_2*nanofibers were characterized by using scanning electron microscopy (SEM), BrunauerEmmettTeller (BET) and X-ray diffraction (XRD). The results indicated that the heat treated* TiO_2*nanofibers consist of anatase and rutile phases. As the calcination temperature increased* $(400-500°C)$, *the anatase phases are greater than rutile phase and specific surface area are decreases while the calcination process influenced the nanofibers diameter.*

Keywords : *Titanium dioxide, nanofibers, electrospinning, calcination temperature*

1 INTRODUCTION

Over the past decades, nanomaterials show the potential for wide ranging industrial, biomedical and electronic applications. It has attracted the attention of many people especially researcher to further research and improve the uses of the nanostructured. Nanostructured such as nanoparticles (Rao and Geckeler, 2011), nanowires (Wu et al., 2012), nanofibers (Wang et al., 2013), and nanotubes (Long et al., 2011) only have size ranging from 1-100 nm. Nanostructured is used as a catalyst in order to improve the process efficiencies because of

the small particles will lead to a greater surface area for the reaction between pollutants andcatalyst (Shen et al., 2014). Due to higher active surface area, nanomaterial is lead to its ability for reducing the toxicity of pollutants to safer level and at a very reasonable cost (Kriklavova and Lederer, 2011). Nanofibers membrane is one of the advanced technologies used because of the small pore size and has very large surface area to volume ratio (Lev et al., 2011). It also has a good flexibility of its surface function and high mechanical performance such as tensile strength (Huang et al., 2003). The excellent features of nanofibers have led to many important applications technology development.

The presence nanofibers have change the fascinating characteristics on the fibers surface such as flexibility of the surface and also providing a very large surface area to volume ratio (Fridrikh et al., 2003). There are several techniques to produce nanofibers such as melt blowing, forcespinning and electrospinning. Heated air blows were used to produce nanofibers in melt blown process (Ellison et al., 2007) while forcespinning technique using the centrifugal forces to turn the material into nanofibers (Padran et al., 2013). Electrospinning is the simplest and attractive method in order to produce fibers which is the diameters is about 10m to 10nm simply by accelerating a jet of charged precursor solution in an electric field (Nor et al., 2013,Vonch et al., 2007). This paper discussed about the physical properties of the prepared TiO2nanofibers which were characterized by SEM, BET and XRD.

2 EXPERIMENTAL PROCEDURE

2.1 Precursor Solution

In this study, polyvinylpyrrolidone (MW$\sim$ 1,300,000, PVP), titanium tetraisopropoxide (TTIP), ethanol and acetic acid from Sigma-Aldrich were used as the starting materials. PVP/ethanol was prepared by mixing 12wt% of PVP to ethanol and was stirred about 2 hours until dissolved. On the other side, TTIP and acetic acid was mixed using ratio of 1:2 of TTIP /acetic acid and stir for 1 hour. TTIP/acetic acid solution was then added to the PVP/ethanol solution and vigorously stirred prior to the TiO_2nanofibers precursor preparation. The basic experimental procedure was discussed elsewhere in which there were some modifications on the original experimental conditions (Liu et al., 2012, Park and Wim, 2009).

2.2 Electrospinning

The precursor solutions were loaded into the plastic syringe with the 21-gauge needle made from stainless steel. The syringe was placed onto the syringe pump with a constant flow rate of 1.0 mL/h and the stainless steel needle was connected to the high voltage supply. The voltage supply is set at 15-20kV and a piece of flat aluminium foil was connected to the ground at 15-20cm distance from the needle tips as the nanofibers collector. The electrospinning process was carried out at room temperature. The obtained as-spun nanofibers were left in the ambient air for one day. In order to remove the polymer content, the as-spun TiO2nanofibers was undergo the heat treatment process. The calcination parameters were varied to identify the effect of different heat treatment process on the TiO_2nanofibers morphology.

2.3 Calcination

During the calcination of the TiO_2/PVP nanofibers, the PVP which is as an organic phase was selectively burned out while the remaining TiO_2 is evolved into polycrystalline. The diameter of the nanofibers will reduce due to the removal of the PVP and sintering the TiO_2 phase. The calcination will take place in $400°C$ and $500°C$ for 3 hour to remove the PVP. The most important characteristics in the nanofibers such as the phase and surface roughness in which they can be controlled by adjust the temperature and the calcination time.

3 RESULT AND DISCUSSION

Figure 1 shows the SEM images of the TiO_2nanofibers before and after undergone heat treatment at different temperature for one hour. Fibers without the calcination process showed a smoother and homogeneous surface because it still contained the polymer binder which is PVP (Li and Xia, 2003). The average diameter of the as-spun nanofibers without calcination was approximately between 200 nm to 1m. In contrast, after the heat treatment it can be seen that the nanofibers shrinking and the diameter became smaller which is the average diameter was about 100nm to 400nm. As the temperature increased, the average diameter of the calcinednanofibers became smaller because of the decomposition of the organic components that presents in the nanofibers (Hu et al., 2013,Nuansing et al., 2006).

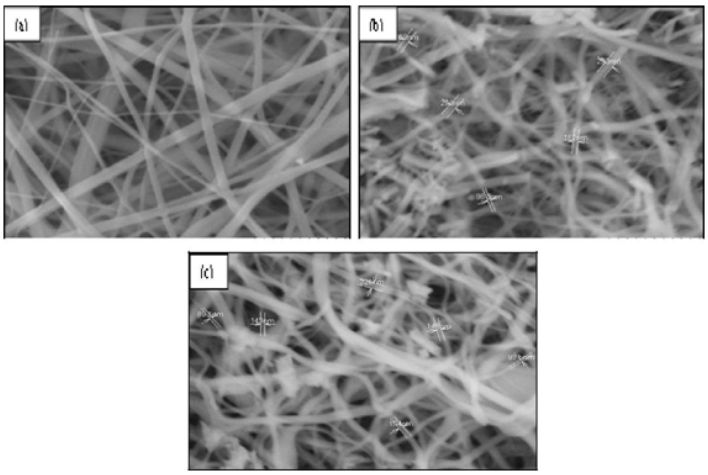

Figure 1: SEM images of TiO2nanofibers a) before calcination and at different calcination temperature b) $400°C$ and c) $500°C$

The BrunauerEmmettTeller (BET) surface analysis of the sample were examined using nitrogen gas with the composition of 30% N2, 70% He. The analysis were conducted for one hour at room temperature ($24°C$) with the outgas temperature is ($130°C$). The specific surface area characterized by BET as a function of calcination temperature was illustrated in Table 1. It was

found that as the calcination temperature increased, the specific surface area of the TiO_2nanofibers decreased. This was due to the increasing in the particle size of the fibers and also the degree of agglomeration occurring in the material is increase (Gaber et al., 2014).

Table 1: Single point surface area data by using nitrogen gas by BET

Sample	Calcination Temperature($^{\circ}C$)	Surface Area (m2/g)
1	400	52.32
2	500	44.17
3	600	8.93

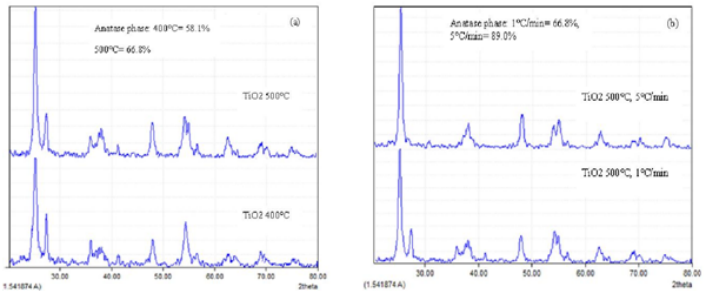

Figure 2: XRD patterns of TiO_2nanofibers at different calcination temperature a) T=400$^{\circ}C$, 500$^{\circ}C$ and different heating rate b) T=5500$^{\circ}C$ (1$^{\circ}C$/min, 5$^{\circ}C$/min)

XRD patterns of TiO_2nanofibers at different temperature and heating rate of heat treatment are shown in figure 2. From figure 2(a) and 2(b), XRD patterns exhibit strong diffraction peaks at 25°, 38° and 54°indicating that the TiO_2nanofibers is in the anatase phase. Meanwhile, strong diffraction peaks at 27°, 36° and 55° indicate that the TiO_2nanofibers is in the rutile phase (Thamaphat et al., 2008, Hanaor and Sorrell, 2001) . Figure 2 also shows that as the calcination temperature slightly increase, the diffraction pattern peak intensity that corresponding to the anatase phase was increased. The prepared sample showing the peaks characteristics of both anatase and rutile phase of the TiO_2nanofibers. Based on figure 2(a), the temperature of the calcination increased from (400$^{\circ}C$ to 500$^{\circ}C$), the percentage of the anatase phase was also increased. The percentage of the anatase phase comparisons was made among the nanofibers after heat treated at 500C with the different heating rate (see figure 2(b)), it can be seen that the anatase phase is greater with the higher heating rate. It can be concluded that heating rate of calcination process an important role in producing highly pure anatase phase of TiO_2.

4 CONCLUSION

TiO_2nanofibers were prepared successfully via simple and efficient electrospinning method by using PVP and TTIP as started materials and heat treated under different temperature. The experimental results showed that smoother and homogeneous surface of TiO_2nanofibers were formed before undergone the calcination process. Based on SEM images, the diameter of the nanofibers is decreasing upon heat treatment process. BET specific surface area analysis showed that the specific surface areas of the nanofibers were decreased with the increasing calcination temperature. XRD analysis have proved that both anatase and rutiles phase have

been obtained after the heat treatment.

5 ACKNOWLEDGEMENT

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