Preparation and Characterization of TiO_2 Electrospun Nano Fibres

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***Abstract* - Titanium dioxide nanofibres were fabricated by home-made horizontal and vertical experimental electrospinning set-ups. Electrospun titanium dioxide (TiO_2) nanofibres were prepared by calcined titanium isopropoxide (TiP) and 2-Methoxyethanol ($CH_3OCH_2CH_2OH$) with different molar ratios and it was formed on Al-foil substrate by different spinning time. TiO_2 sol solution (Sol-gel) was characterized by TG-DTA, XRD and SEM analyses. SEM images revealed that Anatase TiO_2 as-spun fibres on the Al foils were found to be between 120-250nm in diameters before and after calcinations with horizontal set-up for 30 minutes and vertical set-up for 40 minutes spinning time.**

***Keywords* - Electrospinning technique, Nanofibres, TG-DTA, XRD and SEM**

I. INTRODUCTION

Titanium dioxide (TiO_2) is recognized as one of the most promising photocatalystic materials. It has been exploited to be used in extensive environmental protection procedures, for example, air purification and water disinfection [1]. Many methods have been proposed to produce TiO_2 particles, such as sol-gel, hydrothermal, pyrolysis and chemical precipitation. Compared to these methods, sol-gel is by far the most powerful process to produce nanocrystalline TiO_2 with high chemical purity and possess small crystallite size. The properties of TiO_2, especially the catalytic properties, have proved to be strongly related to its crystal structure, morphology and crystallite size [2] ,[6]. One-dimensional (1D) nanostructure materials, such as nanofibers, have received great interest due to high surface area to mass or volume ratio [3], [4], [5].

II. EXPERIMENTAL PROCEDURE

A. Sample Preparation

Titanium isopropoxide (TiP) and 2-Methoxyethanol were chosen as the starting chemical and solvent. Contents of 1:2 Wt% and 3:4 Wt% were used for these materials. Then both materials were mixed together to get the homogenous precursor sol-gel by magnetic stirrer for 6 h. Meanwhile, a few drop of acetic acid was put in it to attain the transparent precursor solution. After that the dehydration process at 90°C was performed to get the viscous gel. Some drops of distilled water added to help controlling the viscosity of this sol-gel. The growth procedure for TiP/2-Methoxyethanol composite is illustrated in Fig 1.

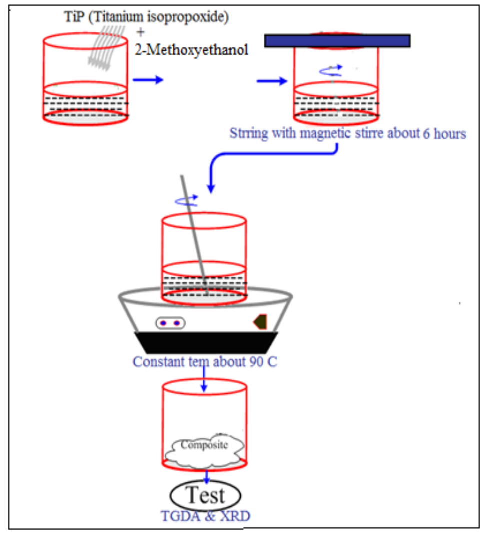


Fig 1. The growth procedure for TiP/2-Methoxethanol composite

B. Mechanism of home-made Electrospinning

Home-made electrospinning machine contained a needle or spinneret, high voltage power supply, and a grounded collector. Horizontal and vertical setups experiments had been done for this research. The operating high voltage power supply was about 25-30 kV. A syringe holder and a collector were kept in Plexiglas tube of cylindrical shape with length of 36.3cm and inside diameter was 8.45 cm, 12 cm and 10 cm apart from each other in horizontal and vertical setups. The positive terminal of dc voltage generator was connected with hypodermic needle (0.55 × 25 mm) and the circular shape of Al collector which was covered with Al foil is connected by negative terminal of Power Supply at ground as shown in Fig 2.

The electrospinning process took place in a cylindrical shape glass tube. There are basically three components such as high voltage supplier, capillary tube and a metal collecting screen. In the electrospinning process, a high voltage was used to create an electrically charged ejecting of polymer solution or melt out of the pipette. Before reaching the collecting screen, the ejected solution was evaporated or solidified, and collected as an interconnected web of small fibres. One electrode was placed into the spinning solution and the other attached to the collector. The collector was simply grounded. High voltage was necessary to generate the electrospinning machine. In high temperature, molten polymers could also be made into nanofibres through electrospinning. The capillary tube, the traveling of the charged fluid jet, and the metal collecting screen must be encapsulated within a vacuum.

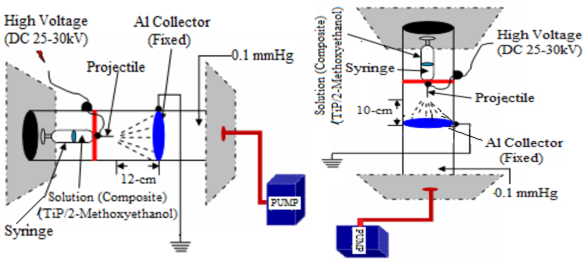


Fig 2.The operation process of Electrospinning technique with Horizontal (Left) and Vertical (Right) setups .

III. RESULTS AND DISCUSSION

Titanium isopropoxide, 2-Methoxyethanol, acetic acid and distilled water were used as starting reagents for the preparation of TiP/2-Methoxyethanol composite fibres. TiO_2 fibre (patterns and spots) on the Al foil were analyzed by the XRD analysis and results are shown in Fig 3,4, 5. According to these figure it is reveals that the Anatase type of TiO_2 was occurred after annealing at 550°C. There are three types of TiO_2 such as Anatase (at 500°C-600 °C), Brookite (at 600 °C-700 °C) and Rutile (at above 700°C). In this research, TG-DTA analysis also performed and result is shown in Fig 6. It is clear that the exothermic reaction of TiP/2-Methoxyethanol sol-gel occurred at 550°C and the edothermic reaction of TiP/2-Methoxyethanol sol-gel occurred at 142.38°C. Therefore TiP/2-Methoxyethanol composite could give the anatase type of TiO_2. After spinning of TiP/ 2-Methoxyethanol composite on the Al foil with different spinning times in horizontal and vertical set-ups, calcined the samples at the temperature of 550 °C to get rid out of the impurities and strengthen the fibres. Then SEM analysis would be performed to ensure whether the fibers were on it or not. The electric field couldn't pull the solution from the syringe, which is inside the capillary tube, to the collector (Al foil) if time of applying the high voltage to the electrospinning machine is not enough.

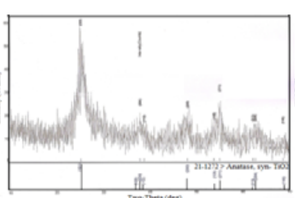


Fig 3. TiO_2 peaks with Anatase library file

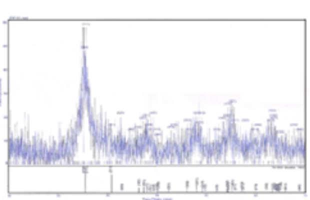


Fig 4. TiO_2 peaks with Brookite library file

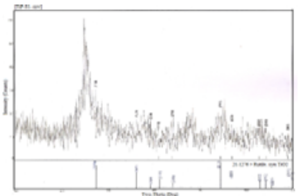


Fig 5. TiO_2 peaks with Rutile library file

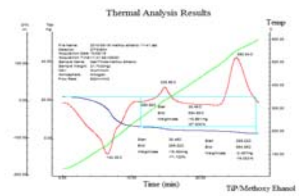


Fig 6. Thermal analysis results for TiP/Methoxyethanol

Some spots which were in place of the fiber patterns on the Al-foil were observed in both set-ups and are shown in Fig7-Fig12. The density or numbers of the as-spun fibers patterns obtained from the vertical set-up were much more compared than that of the horizontal set-up because of the gravitational field. The impinge jet solution with the high electric field strength went easily through the grounded collector (Al-foil) in vertical setup compared than the horizontal setup. The diameters of these spots were measured to be about 100 nm. Moreover, if the vacuum condition was not enough, the electric field due to high voltage on the anode (needle) inside the capillary tube, the solution couldn't go through very well. So some impurities would disturbed the spun solution from the anode (needle) to the collector (Al-foil). Before calcinations, some fibres which were about 120-200nm in diameters were formed on the aluminum surface (Fig10, Fig 11, Fig12) with the spinning time of 30 minutes (horizontal setup) and 40 minutes (vertical setup). After calcination, the size of these fibres was increased to 200-250nm in diameters (Fig 13, Fig 14). In fact, some molecules in the fibres on the foil received more energy when the fibre sample was calcined at high temperature, they would release from the original state or less strengthen the fibre compared to that of before calcinations. The sizes of the fiber pattern also depended upon the electric field strength. If the electric field strength was high enough (above 30 kV) the size became smaller and smaller compared to that of the low electric field strength. But the spinning time was also important. The spinning time should be longer and longer according to these results.

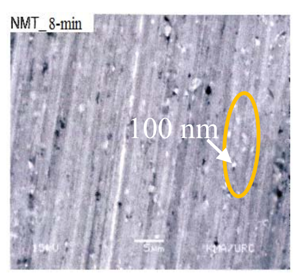


Fig. 7

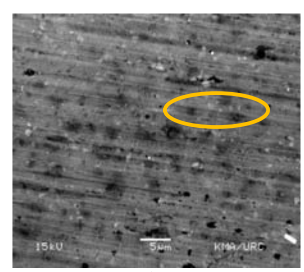


Fig. 8

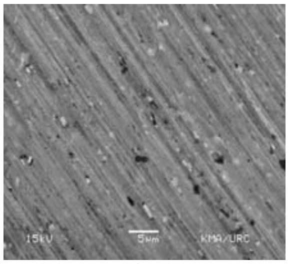


Fig. 9

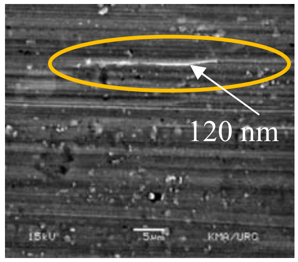


Fig. 10

Fig 7, Fig 8, Fig 9. TiO_2 spots and TiO_2 fibre patterns on the aluminum surface at 8 minutes, 10 minutes and 20 minutes spinning time in horizontal setup before calcinations.

Fig 10. TiO_2 spots and TiO_2 fiber patterns (~120nm in size) on the aluminum surface at 30 minutes spinning time in horizontal setup before calcinations.

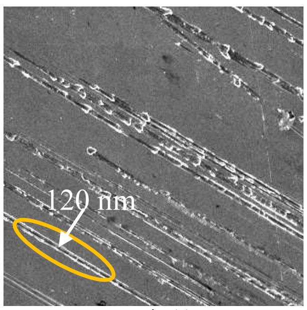


Fig 11

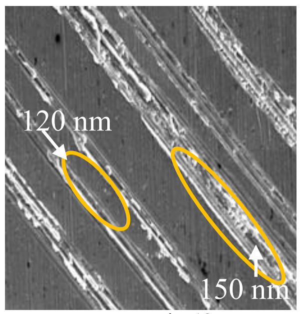
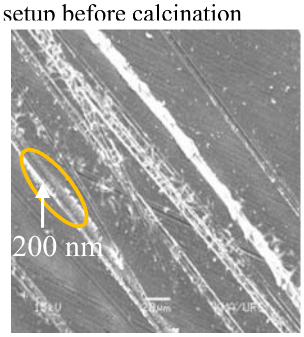


Fig. 12

Fig 12,Fig 13. TiO_2 fiber patterns (~120-150 nm in size) on the aluminum surface at 40 minutes spinning time in vertical setup before calcination



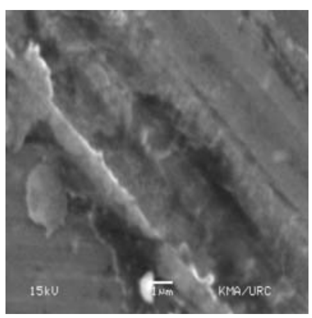


Fig. 13

Fig. 14

Fig 13, Fig 14. TiO_2 fiber patterns (~200 nm in size) on the aluminum surface at 30 minutes spinning time in vertical setup before calcinations (Left) Zoom out (Right) Zoom in

IV. CONCLUSION

TiO_2 fibre patterns and spots have been produced by TiP/2-Methaxyethanol composite sol-gel by home-made electro-spinning technique with horizontal and vertical setups. The XRD and TG-DTA analysis revealed the anatase type of TiO_2. Before calcinations, the sizes of TiO_2 fiber patterns on the aluminum foil is ~120-150 nm by SEM image at 30 minutes (horizontal setup) and 40 minutes (vertical setup) spinning time and spots size also is nearly 100 nm at 22s, 8minutes, 10 minutes, and 20 minutes, respectively. However, after calcining the temperature at 550 C, the size of the fibres increases about 50 nm (i.e ~200 nm in diameter) in both set-ups. Therefore, long electrospinning time, high vacuum condition and suitable calcining temperature are necessary for good and prime-grade nano fibre patterns. Moreover, the density or numbers of the obtained as-spun fibre patterns and the orientation of the fibre mophology from the vertical set-up are comparatively better than those obtained from the horizontal set-up due to the gravitational field and the weight of the sol-gel that is upward from the collector. The impinge jet solution with the high electric field strength therefore will go easily through the grounded collector (Al-foil) in vertical setup and thus it is more feasible for nano fibre fabrication.

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