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Research paper

Fabrication and Characterisation of Titanium Dioxide (Tio$_2$)/PVA Nanofibre Composites using Electrospinning

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Abstract

A study on the formation of titanium dioxide (TiO_2)/ polyvinyl alcohol (PVA) nanofibres has been investigated in the research work. In the study, the TiO_2/PVA solutions were prepared at different concentrations ranging from 1wt% to 10wt%. The PVA solution without TiO_2 was used as control sample in the study. Each solution was extruded using electrospinning at different voltages to form long and continuous nanofibres. The fibres were then characterised for morphological structures, fibre diameter and membrane area. The result shows that control sample of PVA nanofibres formed beads on the fibre surfaces. The addition of TiO2 in PVA was found to reduce the bead formation. The clumps of TiO2 nanoparticles were observed and were also confirmed with the EDX mapping. In addition, the electrospinning parameters affect the formation of TiO_2/PVA nanofibers. At 15 kV, the resultant fibre diameter increases from 153 ± 23 nm to 191 ± 26 nm when the TiO2 concentration was further increased from 1wt% to 10wt%. The morphological structure of TiO_2/PVA nanofibres varies depending on applied voltages and concentrations used. The membrane area of TiO_2/PVA nanofibers is approximately 187 cm^2 for 1wt% of TiO_2. From the study, it shows that an optimal concentration to produce TiO_2/ PVA nanofibers is 10wt%. At 10wt%, no beads were observed on the fibre and the TiO_2 nano-particles were uniformly distributed on the membrane surfaces.

Keywords: *Titanium dioxide (TiO_2) nano-particles;polyvinyl alcohol (PVA); electrospinning; morphological structures; concentration..*

1. Introduction

Titanium dioxide (TiO_2) is a powder-based chemical which is low cost, chemically stable and non-toxic [1]. In the past, titanium dioxide has been studied extensively in a broad range of industrial and scientific field [2]. Titanium dioxide (TiO_2) has many interesting properties such as high photo activity, self-cleaning and antimicrobial which leads to receive much attention from researchers [3]. TiO_2 used to be applied on different substrates such as activated carbon, glass, stainless steel [4] and silica [5]. However, the use of TiO_2 for conventional separation method was rather difficult and it becomes the drawback for the wide application of TiO_2 [6]. In this study, the TiO_2 nanoparticles have been bounded into composite nanofibres using polyvinyl alcohol (PVA) as fibre template throughout electrospinning process.

Electrospinning is a facile and straightforward process that uses a high-voltage electric field to produce ultrafine nanofibres [7]. Electrospinning is basically operated by using the concept of electrostatic that impart towards polymer solution in order to produce fine fibres with the range of 100nm-500nm. According Mirjalili *et al*. [8], electrospinning occurs when the polymer solution is subjected to certain heat and forced to form droplet at the tip of syringe. Then, high voltage applied to polymer solution to aid the polymer acceleration on the surface of collector in order to form nanofibers. The accumulation of the nanofibre forms a porous membrane.

Polyvinyl Alcohol (PVA) is one of synthetic polymers that can be spun using electrospinning. PVA nanofibres produced from the electrospinning technique have gained a great deal of attention due to their unique contribution of properties such as highly hydrophilic, small pore sizes, fine fibre diameter, light weight and high surface area [9-12]. Due to these properties, the PVA nanofibres have potential to be employed in various applications such as filtration [8-9] and biomaterials [9-12]. In addition, the PVA can be incorporated with any inorganic materials such as titanium dioxide (TiO_2), carbon nanotube (CNT) etc. Therefore, the aim of the study is to investigate the spinnability of TiO_2/PVA solution using electrospinning. This research study focuses on identifying an ideal concentration of titanium dioxide (TiO_2) and polyvinyl alcohol (PVA) for the formation of nanofibre composite as well as characterizing fibre morphology, fibre diameter and membrane area for titanium dioxide (TiO_2)/polyvinyl Alcohol (PVA) nanofibre composite.

2. Materials and method

2.1. Materials

Polyvinyl alcohol (PVA) (Sigma-Aldrich, Mw 125,000 g/mol) and titanium dioxide (TiO_2) nano-particles (Sigma-Aldrich) were used as received. Distilled water was used as solvent in the experiments.

2.2. Preparation of control PVA and $Ti0_2$/PVA solutions

PVA and TiO_2 nano-particles were dissolved in distilled water at 120°C for 4 hours. The concentration of TiO_2 was in a range of 1wt% to 10 wt%. The solutions were left for 24 hours at room temperature electrospinning process. The preparation of control PVA solution was briefly described elsewhere [9].

2.3. Electrospinning process

Fig. 1 illustrates the fabrication of control PVA and TiO_2/PVA nanofibre composites using electrospinning. In this study, the control PVA and TiO_2/PVA solutions were electrospun at constant distance of 10cm with 0.5ml/hr of flow rate. Each solution was electrospun at different applied voltages ranging from 10kV to 15k. The control PVA and TiO_2/PVA nanofibres composites were collected onto grounded aluminium foil for about 5 minutes. The resultant fibres and membranes were characterized for morphological structure, fibre diameter and membrane area.

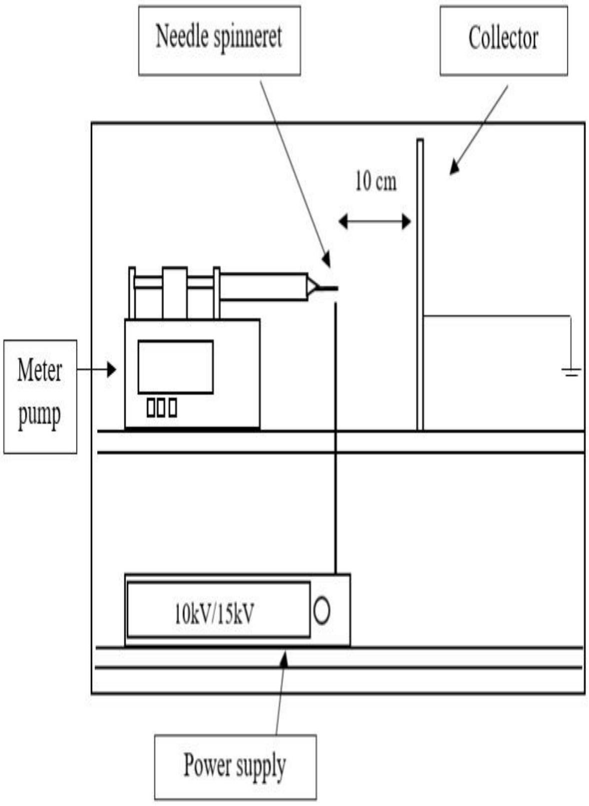

Fig. 1: Schematic diagram of electrospinning process.

2.4. Fibre and membrane characterization

For fibre diameter, 50 measurements were taken at random places and were measured using ImageJ software.

For membrane area, the fabricated membrane was scanned over a transparent grid with an area of $1cm^2$ per grid. The scanned image was then measured using ImageJ software. Three measurements were taken for each sample.

The presence of TiO_2 nano-particle on fibre surface was tested using EDX mapping.

3. Results and discussion

3.1. Effects of TiO_2 concentrations on the morphological structure of TiO_2/PVA nanofibres composites.

Figs. 2 (a)-(d) illustrate the formation of control PVA (polyvinyl alcohol) and TiO_2/PVA nanofibre composites using electrospinning**.** From the results, it shows that control PVA and 1wt% TiO_2/PVA nanofibre composites have long and continuous fibres with beads on the fibre surfaces (Figs. 2 (a)–(b)).

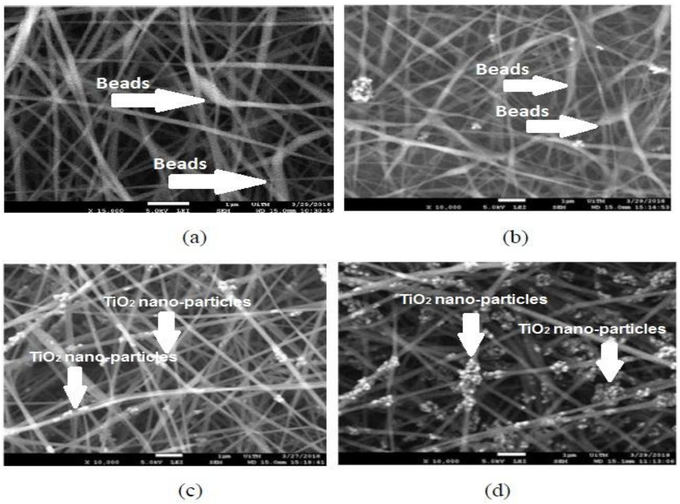

Fig. 2: Typical SEM images of (a) control sample PVA, (b) 1wt% TiO_2/PVA, (c) 5wt% TiO_2/PVA and (d) 10wt% TiO_2/PVA, respectively.

The formation of beads was probably due to low viscosity of solution for control PVA and 1wt% TiO_2/PVA. At lower viscosity, the solution was insufficient to overcome high columbic repulsion force during electrospinning process, which resulted in beads on fibre surfaces. Similar finding was observed by Zhang *et.al* [10], where they reported that solution with low viscosity produced beaded fibres.

As the concentration increases from 5wt% to 10wt% the amount of beads reduce (Figs. 2 (c)-(d)). This indicates that solution with high viscosity has sufficient polymer chain entanglement that able to withstand high columbic repulsion force during electrospinning. The result was in agreement with Zhang *et.al* [10]. They observed the transition from beaded fibres to uniform cylindrical fibres as the PVA viscosity increased [10].

In addition, the TiO_2 nanoparticels are well distributed as the concentration increases from 5wt% to 10wt%. This also shows that solution with high concentration of TiO_2 (10wt%) disperses more TiO_2 nanoparticles on fibre surfaces than solutions at 1wt% and 5wt%.

The presence of the TiO_2 nanoparticles was analyzed using EDX mapping. As shown in Fig. 3, the Ti peaks prove the existence of TiO_2 nanoparticles on the fibre surfaces. Al peaks indicates backing material of the nanofibres.

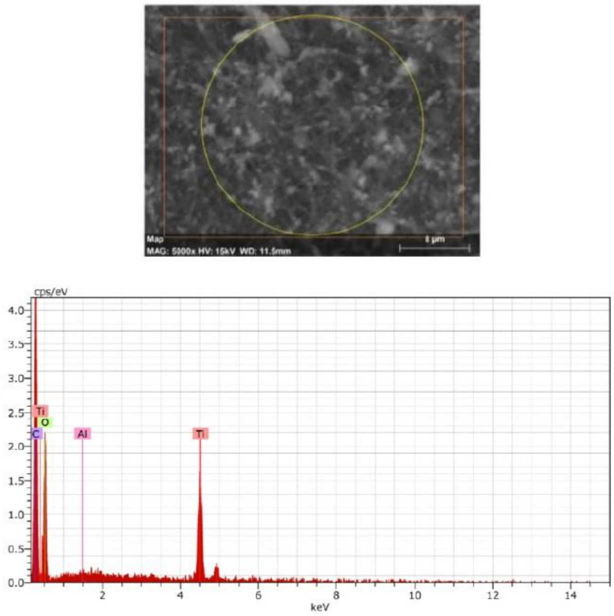

Fig. 3: Typical EDX mapping for TiO_2/PVA nanofibres.

3.2. Effect of applied voltage on morphological structure and fibres diameter of control sample PVA and TiO_2/PVA nanofibres.

Figs. 4 (a)-(h) depict the effect of applied voltages on morphological structures of control PVA and TiO_2/PVA nanofibre composites. The increase of applied voltages produced beaded fibres for control PVA and 1wt% TiO_2/PVA. As mentioned earlier, the formation of beaded fibres was due to the low solution viscosity where the solution was insufficient to overcome high columbic repulsion force during electrospinning process.

For 5wt% and 10wt%, cylindrical fibres with TiO_2 nano-particles were fabricated at any voltages. This indicates that solutions at 5wt% TiO_2 and 10wt% TiO_2 have sufficient polymer chain entanglement that able to withstand high columbic repulsion force during electrospinning. As reported Zhang *et.al* [10], the transition from beaded fibres to uniform cylindrical was due to the increase of PVA solution concentration [10].

As shown in Fig.5, the increment of voltages from 10kV to 15kV reduced the control PVA fibre diameter from 178 + 29 nm to147 + 23 nm. It was expected that an increase in applied voltage gave higher elongation to the polymer jet which resulted in the reduction of fibre diameter. The reduction of fibre diameter at higher voltage was also observed by other recent studies [9-11]

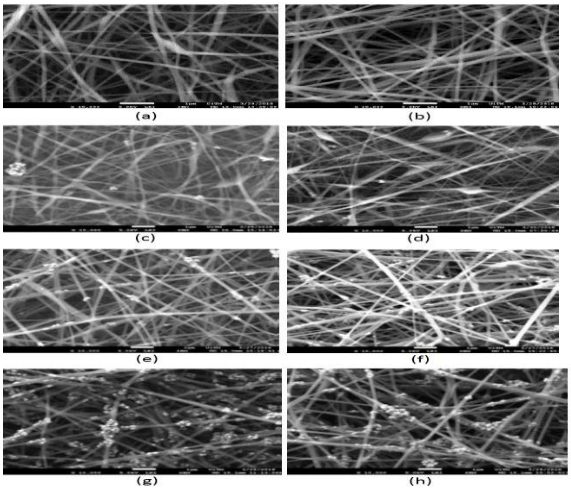

Fig. 4: Typical SEM images of control sample PVA at (a)10kV and (b) 15kV, 1wt% TiO_2/PVA nanofibres at (c) 10kV and (d) 15kV, 5wt% TiO_2/PVA nanofibres at (e) 10kV and (f) 15kV and 10wt% TiO_2/PVA nanofibres at (g) 10kV and (h) 15kV.

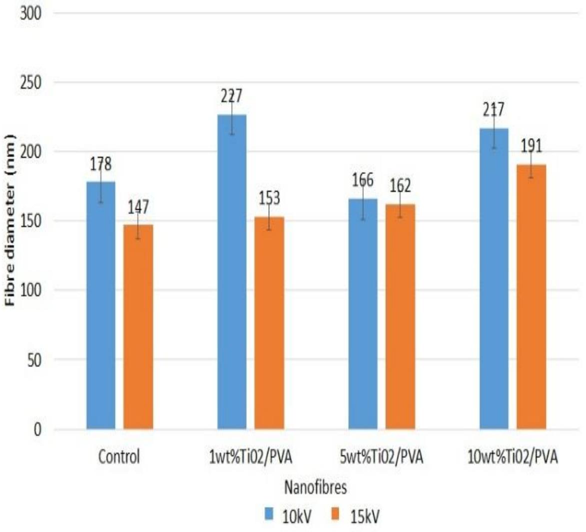

Fig. 5: Average fibre diameter against control sample PVA and TiO_2/PVA nanofibre composites at 10kV and 15kV.

For 1wt%TiO_2/PVA nanofibres, it was found that the diameter decreased from 227 + 29 nm to 153 + 23 nm with increasing of applied voltage. Similar result was also found for 5wt%TiO_2/PVA nanofibre composites, where the fibre diameter reduced from 166 + 29 nm to 162+ 23 nm with increasing of applied voltage from 10kV to 15kV. The reduction of fibre diameter was also found for 10wt%TiO_2/PVA. As shown in Fig.5, the fibre diameter of 10wt%TiO_2/PVA nanofibre composites reduces from 217 + 23nm to 191 + 28nm when the voltages decrease from 10kV to 15kV. Recent studies reported that the reduction of fibre dimeter was due to the further elongation of fibre at higher voltages [9-11]. In addition, Khajavi and Damerchely [12] reported the resultant PVA nanofibres diameter was dependent on voltage and concentration used.

3.3. Effects of applied voltage on membrane area

Table 1 tabulates membrane area for control PVA and TiO_2/PVA nanofibre composite. The membrane area of control PVA was found to increase from 127 ± 1.5 cm^2 to 185 ± 1.3 cm^2 with increasing of voltages. When a higher voltage was applied, jets with bigger bending instabilities could form. As a result, a large membrane area was deposited onto collector. The results were in agreement with Affandi NDN [11], where the author observed the increase of membrane area at higher voltage.

However, for TiO_2/PVA membrane, the membrane area decreases when the voltages increase from 10kV to 15kV. As shown in Table 1, the membrane area of 1wt% TiO_2/PVA nanaofibre composites reduces from 187 ± 1.2 cm^2 to 123 ± 3.0 cm^2. The membrane area for 5wt% TiO_2/PVA and 10wt% TiO_2/PVA also reduced with increasing of applied voltage ranging from 10kV to 15kV. It was expected that the presence of TiO_2 in solution produced unstable charged jet at higher voltage. This has resulted in a small membrane area for TiO_2/PVA nanofibre composites. Similar finding also observed by Supaphol *et. al* [13], where the authors reported that an increase in applied voltage reduced the area of electrospun nanofibre membrane.

Table 1: Area of control PVA and TiO2/PVA nanofibre membranes

Applied voltage (kV)	Area of membrane (cm^2)			
	Control sample PVA	1wt% TiO_2/PVA	5wt% TiO_2/PVA	10wt% TiO_2/PVA
10	127 + 1.5	187 + 1.2	180 + 1.4	186 + 1.5
15	185 + 1.3	123 + 3.0	177 + 2.6	169 + 1.2

4. Conclusion

The incorporation between PVA and titanium dioxide were investigated and the optimal TiO_2/PVA concentration was identified in the study. The result shows that the optimal concentration for TiO_2/PVA solution was 10wt%. At 10wt%, no beads were observed on the fibre and the TiO_2 nano-particles were uniformly distributed on the membrane surfaces. The solution concentration also affects morphological structures of TiO_2/PVA nanofibre composites. The control sample of PVA nanofibres formed beads on the fibre, whereas the TiO_2/PVA nanofibre composites produced cylindrical fibres. The deposition of TiO_2 nano-particle on the fibres was clearly observed and was confirmed with the EDX mapping. At 15kV, the resultant fibre diameter was found to increase from 153 ± 23 nm to 191± 26 nm when the TiO_2 concentration increased from 1wt% to 10wt%. In addition, the membrane area of TiO_2/PVA nanofibre composites decrease with increasing of applied voltage. From the experimental works, it shows the feasibility to form TiO_2/PVA nanofibre composites using electrospinning. Further research will be focused on the potential applications of the TiO_2/PVA nanofibres.

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