



## Electrospinning of Polyamide Fiber containing Nano TiO<sub>2</sub> and the Effect of Heat, Setting on Self-cleaning

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### ABSTRACT

This paper reports the results of an investigation aiming at finding what affect the nano TiO<sub>2</sub> powder in electrospinning of polyamid66. PA66 chips with 0.5% nano TiO<sub>2</sub> was solved in formic acid and prepared for electrospinning. Spinning have been done on surface of Nylone66 fabric. Half of the treated fabric was heat setted and the other half wasn't. Then all samples were stained with dye and the photocatalytic activity of nano TiO<sub>2</sub> has been examined under UV irradiation. Their self-cleaning property was investigated by reflectance spectrophotometer. Also SEM and TEM of nano TiO<sub>2</sub> with XRD test were helped to better analyses of the results.

**Key words:** Nano TiO<sub>2</sub>, PA66, Self-cleaning, Photocatalytic, Electrospinning.

### INTRODUCTION

Polymer nano fibers have gain much attention because of their fundamental importance in a wide range of applications such as filtration, tissue industry, medical science, drug carrier, biomedical applications and conductive yarns<sup>1-5</sup>. Reduction of polymer fiber diameter in to nano scale gives rise to a set of desirable properties including an increase in surface-to-volume<sup>6,7</sup>, controlled release of drugs<sup>8</sup>, high anisotropic electrical conductivity<sup>8,9</sup>, and enhanced light scattering and photoluminescence<sup>10</sup>.

Based on earlier research results, electrostatic fiber spinning (electrospinning) is a

technique using electrostatic force to produce inexpensive fine filaments<sup>11</sup>. Spinning of solution polymer by using an electric filed can be traced back to the early 1930s. By electrospinning, nano polymeric fibers can be produce with diameter of about 100nm<sup>12-14</sup>. These fibers have small size, high surface area and higher porosity<sup>11,15</sup>. In electrospinning, polymer solution can be spun into smaller diameter fibers using a high electric potential (10-30Kv). In this process the electric field (at the needle) overcome the polymer surface tension and causes to eject the solution. Then the solvent will evaporate and the fiber aggregate on the collector<sup>16</sup>. In this process some parameters will impress such as temperature and humidity<sup>17</sup>.

Scattering of some nano materials like nano titania ( $\text{TiO}_2$ ) within polymeric fibers (nano composites) can enhance the fiber properties such as conductivity, dyeability and strength. Of course these properties depend on structure and amount of used nano materials<sup>18-23</sup>.  $\text{TiO}_2$  has strong oxidation characteristics, excellent chemical stability and also competitive cost effectiveness<sup>24</sup>. One of the principal applications of nano  $\text{TiO}_2$  is using as catalytic support<sup>23</sup>. The photocatalytic activity of nano  $\text{TiO}_2$  is depending on its phase and nano  $\text{TiO}_2$  has three phases: Anatase, Rutile and Brookite<sup>26</sup>.

Very little research on electrospinning of polyamides (PA) contain nano materials has been performed that has just been focused on the fiber formation and their physical properties. Thus our research focused on selfcleaning of PA fabric that was coated with nano fibers which contains nano  $\text{TiO}_2$  (nano composite) in two kinds of heat set and non-heat set. The goal of this study is investigation the selfcleaning of fabrics (which are coated with nano fibers) and find out best result with/without heat setting.

## MATERIALS AND METHODS

### Materials

The plain weave 100% super bright nylon66 fabric was used with the wrap density of 42 yarn/cm, the weft density of 24 yarn/cm and the number of 75den. Nano titanium dioxide powder (Degussa P-25) with average particle size of about 21 nm from Evonik was employed. Super bright nylon chips from Aldrich (N66 74712) were prepared. Formic acid ( $\text{CH}_2\text{O}_2$ ) as chips solution from Merck was prepared. The using dye for staining was Dark Green.

### Electrospinning

For the preparation of precursor solution of electrospinning, nano titania suspension in steady concentration (0.5%) was sonicated for 30min at 50°C in bath of N66 chips/formic acid. A sheet of N66 fabric was putted on the device collector and then electrospinning was beginning. Spinning potentials ranged from 14 to 25 kV with pumping rate of 0.3ml/h and the distance was 20cm. Thus nano fibers contain nano  $\text{TiO}_2$  was collected on fabric base.

### Heat set and Fixation

In order to fixation of nano web on PA fabric, half of the samples were putted in an oven for 2 min at 80°C. Therefore, we had two kinds of samples: heat setted and non-heat setted. In the next, the effect of heat setting will be investigate and compare with results of the other samples.

### Staining and irradiation

All samples were stained with Dark Green (CI 30295) and were irradiated under UV lamp (Philips, HPA 400 s, Belgium).

### Characterization

XRD were obtained using INEL, model EQUINOX3000, in order to investigation of crystal size and phase of nano material. Also microscopic investigation on fabric samples were carried out using a Philips XL30 (Netherlands) scanning electron microscope (SEM) and Philips EM208 transitions electron microscope (TEM). Reflectance spectrophotometer (BYK Gardner, India, with CIELAB 1976 color space and D65-light source) was used for investigation of selfcleaning.

## RESULTS AND DISCUSSION

### XRD, SEM and TEM analysis

SEM and TEM photos of nano  $\text{TiO}_2$  are illustrated in Fig1&2. Fig3 shows the SEM analysis of electrospun fabric which is containing nano  $\text{TiO}_2$ . As it shown, the heat set sample has more nano electrospun fiber on its surface in compare of non heatsetted. So heat setting has positive effect on its stability. It can be clearly seen that particle distribution is appropriately remarkable especially in heat setted

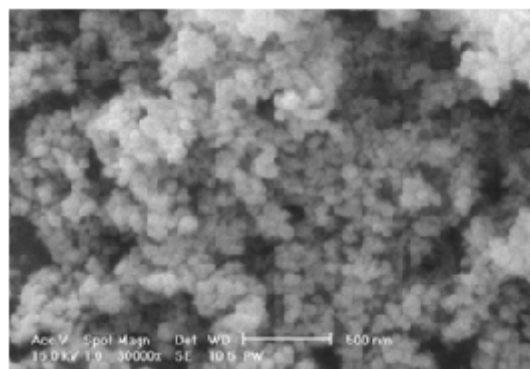
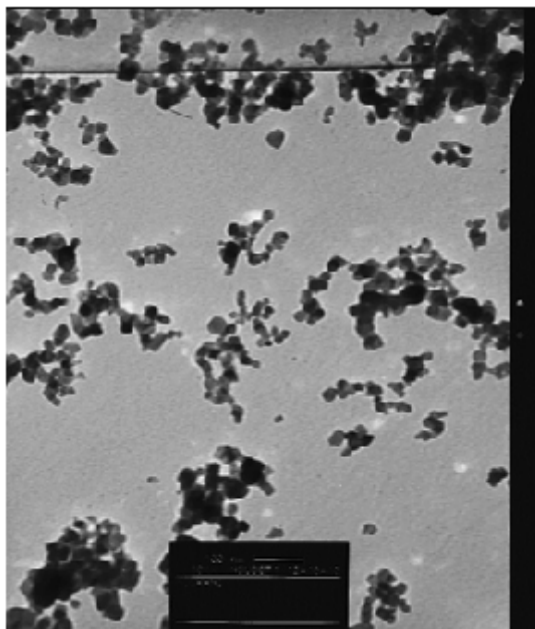


Fig. 1: SEM of nano  $\text{TiO}_2$  powder

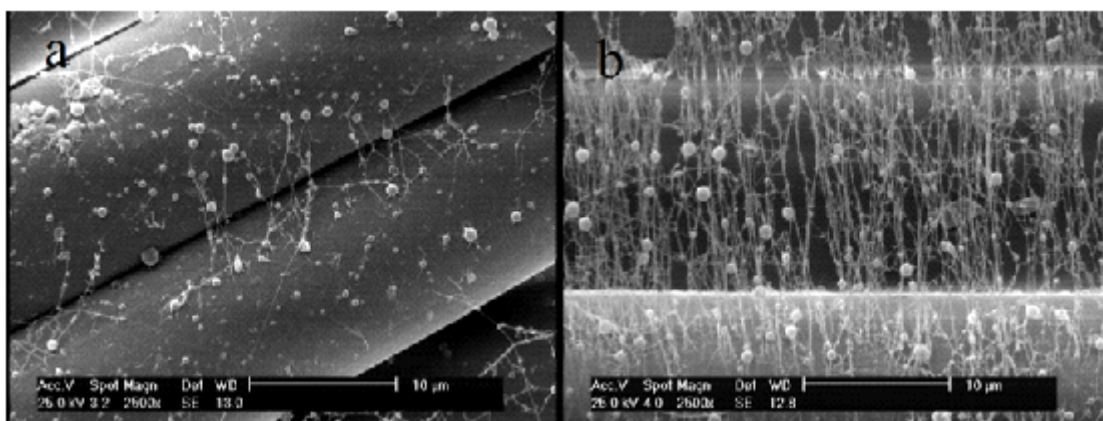


**Fig. 2:** TEM of nano TiO<sub>2</sub> powder

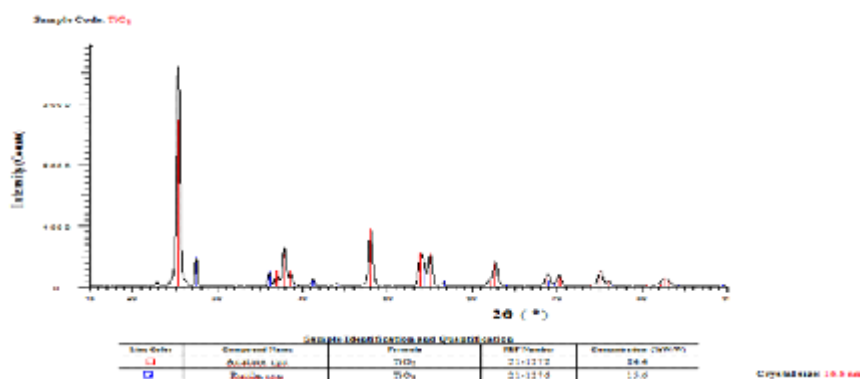
sample. The XRD spectra of the nano TiO<sub>2</sub> is show in Figure4. Two peaks of anatase & rutile observed for P25. Quantification analysis shows that the average percentage of anatase & rutile is 84% and 16 %respectively and the crystal size was 16.6nm.

**Self-cleaning Properties**

The structure of dye that used for staining is shown in figures5. The treated samples and raw fabric (as reference) were stained with thisdye (2drops each sample). After irradiation of 400w UV lamp for 16h at room temperature, the self-cleaning property of fabrics was achieved by ΔE from reflectance spectrophotometer. The results show that using nano TiO<sub>2</sub>disperse in electrospinning solution can improve self-cleaning of fabric. As it shows in diagram6, self-cleaning of heat setted sample is higher than the other. It means that heat setting cause to have more nano fibers and nano TiO<sub>2</sub> in order to selfcleaning. Of course having ΔE



**Fig. 3:** SEM of electrospun fabrics: a) non-heatsetted, b) heatsetted



**Fig. 4:** Spectra of nano TiO<sub>2</sub> and crystal phasepercent

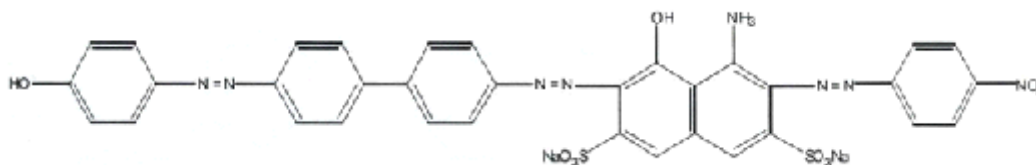
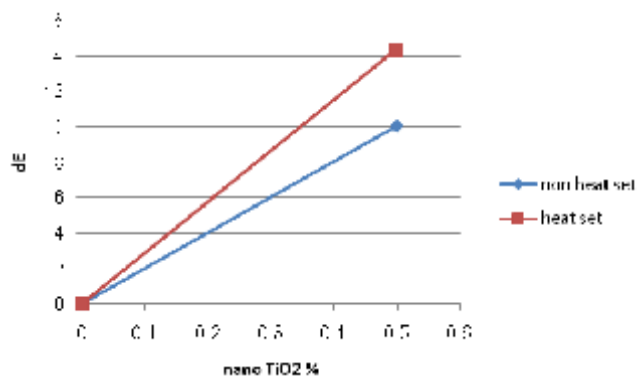


Fig. 5: Structure of Dark Green

Fig. 6:  $\Delta E$  of heat setted & non heatsetted samples

more than 10 show that using nano TiO<sub>2</sub> in electrospinning solution is an effective method. Regarding the SEM photos, highest selfcleaning of heatsetted sample is because of more homogeneous of nano particles on surface of fabricand more nano fibers.

### CONCLUSION

The objective of this study was

investigation of self-cleaning behavior of polyamide66 fiber coated by nano fibers containing nano TiO<sub>2</sub>. The results show that nano TiO<sub>2</sub> dispersed in electrospinning solution give self-cleaning property to fabric. Also heat setting of electrospinning fabric has more selfcleaning property because of more stability of electrospun fibers and nano TiO<sub>2</sub>. In general, using nano TiO<sub>2</sub> in spinning of polyamide6 improve the properties of this fiber and cause to improve the self-cleaning property.

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