

ELECTROSPUN TiO₂ NANOFIBERS AS A MATRIX TO ENHANCE THE DRUG LOADING

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ABSTRACT

The technique of Electrospinning has been considered as the most promising approach for the large scale production of nanofibers of varying diameter. It is quite a simple and efficient method for the nanofiber production. When a viscous fluid is charged with a high voltage, the electrostatic force draws the fluid into a liquid jet. Because of the interaction between the jet and external electrical field and charge repulsion inside the jet, the charged jet undergoes a bending instability to stretch it thinner. Solvent evaporation from the filaments results in solid fibers. It now remains important to explore and expand the scope of applications of as produced nanofibers. We present a simple procedure to load an herb onto the nanofiber. Morphology and size of as-prepared titanium fibers were observed by scanning electron microscope (SEM). The B.E.T method was used for surface area calculation. Crystalline phase of the fibers was identified by X-ray diffraction. Here we loaded the TiO₂ nanofiber matrix with the well-known antibacterial Curcumin drug. We have generated an absorption spectrum of the desorbed Curcumin from the fiber matrix. It is found that the fibers with fewer diameters are having more amount of drug adsorption.

Keywords: Curcumin, Electrospun, Nanofiber matrix, Wound dressing, XRD

I. INTRODUCTION

Electro spinning uses an electrical charge to draw very fine fibers from a liquid [1,2]. When a sufficiently high voltage is applied to a liquid droplet, the liquid becomes charged, and electrostatic repulsion overcomes the surface tension and the droplet is stretched; at a particular point a stream of liquid erupts from the surface. This point of eruption is known as the Taylor cone [3]. If the molecular cohesion of the liquid is sufficiently high, stream breakup does not occur and a charged liquid jet is formed. Solvent evaporation results in solid fibers [4, 5]. The jet is then elongated by a whipping process [6] caused by electrostatic repulsion initiated at small bends in the fiber, until it is finally deposited on the grounded collector. The elongation and thinning of the fiber resulting from this bending instability leads to the formation of uniform fibers with nanometer-scale diameters [7].

Here reports the synthesis of TiO₂ nanofibers as a matrix to load the well known antimicrobial drug Curcumin [8-11]. The morphology of the fibers are studied and the drug loading capacity of the fibers are also studied from the drug desorption studies.

II. EXPERIMENTAL

Electro spinning unit contains a high voltage power supply, a stand to hold the syringe with very small diameter and a collector. In electro spinning process the nanofibres of metal oxides are produced from a liquid droplet of liquid polymeric solution under the influence of electrostatic forces. The droplet overcomes the surface tension force holding the droplet because of the increased electric force and allows the drop to falls on the conductive collector. The high voltage difference between the needle tip and the collector helps the droplet to be drawn in to a fiber in the form of a jet. The fiber diameter and morphology can be changed by controlling the electric field and injection rate. It is also depends on the viscosity of the polymeric solution. In the present study we prepared one dimensional TiO_2 nanofibres with different diameter by changing the flow rate of the polymeric solution

2.1 Preparation of the Polymer Solution

2.5g PVP (Poly Vinyl Pyrrolidone) is added in 50ml ethanol (5wt% ethanol solution of PVP) and is magnetic stirred well at room temperature. Then 27.5ml ethanol, 27.5ml acetic acid and 15.8ml Titanium Tetra Isopropoxide is mixed with the 5wt% ethanol solution of PVP. These two solutions are mixed well for 48 hours.

2.2 Electro Spinning of TiO_2 Nanofiber

The prepared polymer solution is taken in a syringe tube of diameter 15.6mm and placed in the electro spinning apparatus. Cleaned FTO glass plate which is masked by using scotch tape is fixed on the aluminum foil in the apparatus. The distance between the needle tip and the glass plate is set as 14.5 cm. Set the values of duration as 1h and feed rate as 0.5 mL/h, 0.9 mL/h and 1.1 mL/h. By applying proper voltage 12 kV, TiO_2 Nanofiber will start to form on the glass plate.

2.3. Curcumin Loading in Fiber Matrix

Nanofibers of different flow rates, namely Sample 1 (0.5 mL/h), sample 2 (0.9mL/h) and sample 3 (1.1 mL/h) obtained, were cut in specific diameters. 0.3 mM solution of Curcumin in methanol is prepared. The fibers obtained for different feed rate were immersed in this solution overnight (8-16hrs) for drug adsorption.

2.4. Curcumin Desorption from the Fiber Matrix

In order to analyse the drug (Curcumin) loading, drug was desorbed completely from the curcumin- adsorbed films by immersing it in to NaOH ethanolic solution 0.1 mM [12] for 12 h. A spectrophotometer was used to measure the desorbed dye concentration in this solution

III. RESULTS AND DISCUSSION

Electrospinning technique provided fibers of different diameters, that is it generated nanofibers of different flow rates. Among other ceramics our interest lay on titanium dioxide due to its extraordinary electrical, electro chemical and catalytic properties. Since curcumin is a drug having a proven antimicrobial activity we have chosen curcumin as the herb to be loaded onto the fiber matrix.

3.1. TiO₂ Nanofiber Morphology from SEM Image

The fibrous morphology was analyzed before and after calcining. The SEM image of the TiO₂ nanofibers prepared using the polymer solution by electrospinning setup and is obtained before and after calcinations process for all the three samples.

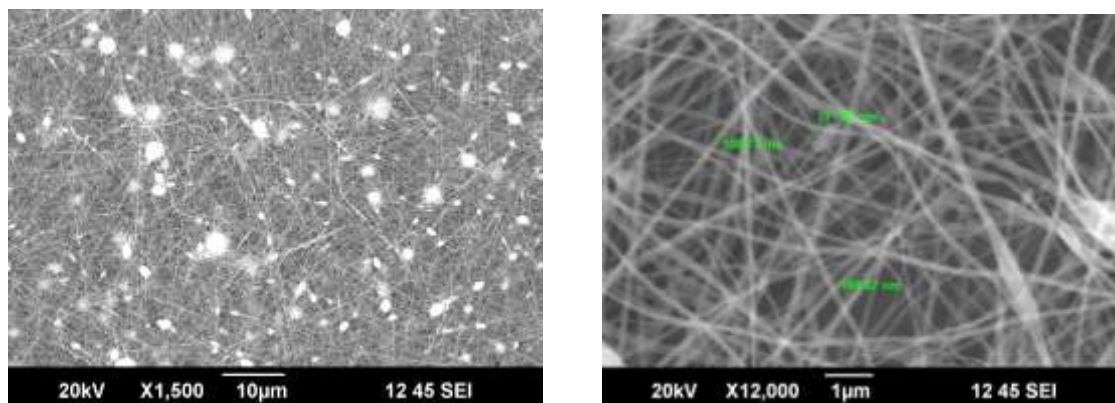


Fig.1: (a)and(b) SEM Images of Electrospun TiO₂ Nanofibers Before Calcination Sample 1

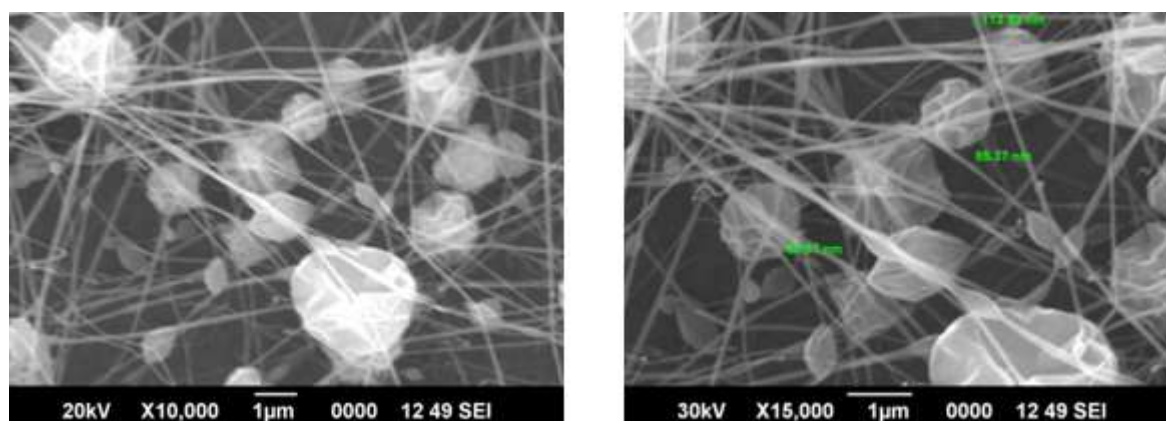


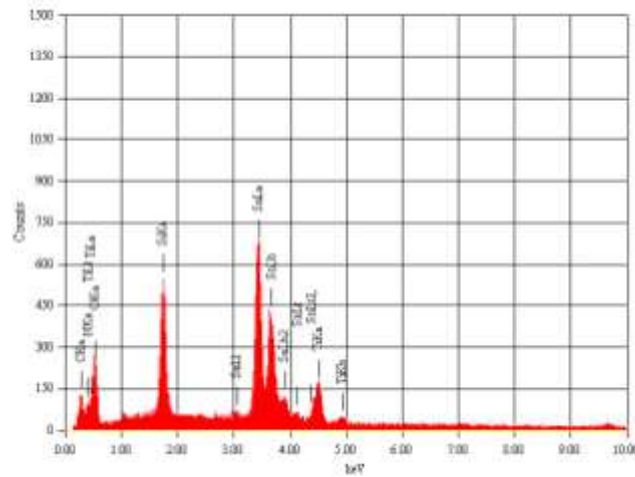
Fig.2: (a) and (b) SEM Images of Electrospun TiO₂ Nanofibers after Calcination Sample 1

The SEM images give a clear picture about the polycrystalline structure of the TiO₂ nanofibers with crystalline size of the fiber diameter ranges from 106 to 166 nm before the calcinations for the sample 1. From the SEM images shown in Figure 1 and Figure 2 , it is clear that the heat treatment does not affected the morphology of the fibers. Moreover the diameter of the fiber size decreases from 106 - 166 nm to 66-113 nm ranges. The reason for this can be explained as the polymer and titanium isopropoxide will undergo decomposition under high temperature it may lead to decrease in fiber diameter. Another reason is that the titanium iso propoxide was uniformly distributed in the PVP matrix. So after the complete pyrolysis process of PVP the phase of TiO₂ was preserved better. The average diameter of all the three samples are given in Table 1

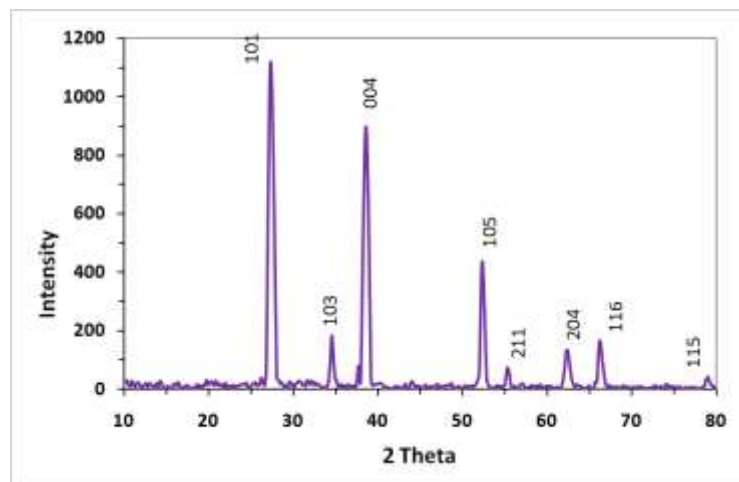
Table 1: The Average Diameter of the Samples

Samples	Feed rate (mL/h)	Average diameter (nm)
Sample 1	0.5	89.5
Sample 2	0.9	95.2
Sample 3	1.1	118.0

The SEM-EDAX image of TiO₂ nanofibers after calcinations confirms the presence of Titanium dioxide (TiO₂). All the peaks are exactly matching with the XRD pattern. This confirms that only TiO₂ is present in the fiber. There are no traces of the polymer PVP.



3.3. XRD Analysis of Calcined Fibers (X-Ray Diffraction)



We have compared the result of XRD data of TiO_2 nanofibers shown in Figure 3.6 obtained by electrospun using polymer solution and titanium isopropoxide in ethanol. It is annealed at 150°C for 1 hour. From the data which is compared with the JCPDS file No 21-1272. It is clear that typical anatase diffraction peaks were only visible. It confirms the formation of pure anatase phase and complete absence of the polymer PVP.

The curcumin drug is completely desorbed from all the three fiber matrix using KOH solution and its absorption spectra are taken using a spectrophotometer. It is shown in figure 3.7. The fiber made from lesser feed rate (0.5 mL/h) is having more drug absorption capacity. It is marked as sample 1 in absorption spectra.

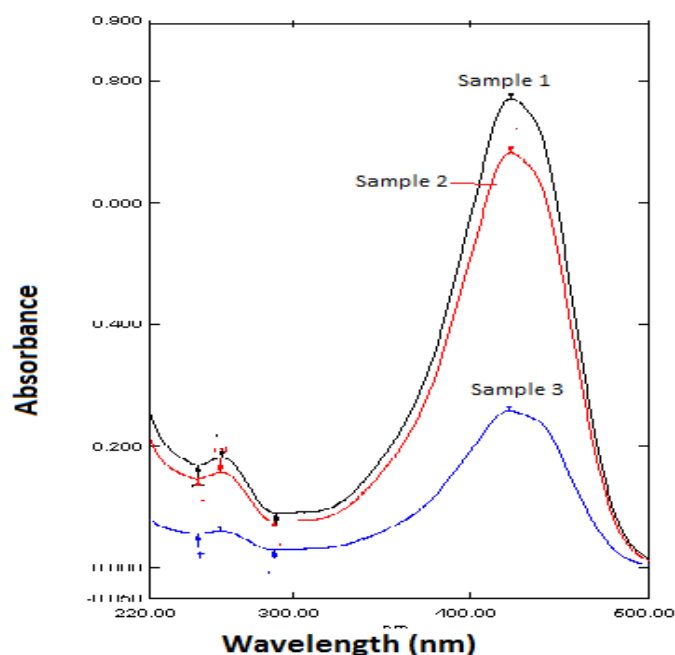


Fig 5: Absorption Spectra of the Desorbed Curcumin

IV. CONCLUSION

In present study, the ability of the Nanospider™ Technology to produce fibrous TiO₂ was demonstrated. Electro spinning has been considered as the most promising approach to produce continuous nanofibers on a large scale and the fiber diameter can be adjusted from nanometers to micrometers. Technically, electro spinning is a process that uses a strong electrical field to draw a polymer fluid into fine filaments. Application of high voltage generates thin layers of nanofibers. We lay our interest in characterizing the TiO₂ nanofibers. Morphology and size of as-prepared titania fibers were observed by scanning electron microscope (SEM). Crystalline phase of the fibers was identified by X-ray diffraction, which is confirmed by SEM EDX. It is found that the sample1 matrix which is having least diameter of 89.5 nm is capable of adsorbing more drug on it because of its larger surface area per centimeter square.

Biological application of TiO₂ nanoiber is that, we can use them as dressing materials. Titanium dioxide as such has antimicrobial activity [13]. Wound healing is a most relevant application of nanofiber. Dressings for wound healing function to protect the wound, exude extra body fluids from the wound area, decontaminate the exogenous microorganism, improve the appearance and sometimes accelerate the healing process [14, 15]. For these functions, a wound dressing material should provide a physical barrier to a wound, but be permeable to moisture and oxygen. Electrospun nanofiber mat is a good wound dressing candidate because of its unique properties: the highly porous mat structure and well interconnected pores are particularly important for exuding fluid from the wound; the small pores and very high specific surface area not only inhibit the exogenous microorganism invasions, but also assist the control of fluid drainage; in addition, the electro spinning process provides a simple way to add drugs into the nanofibers for any possible medical treatment and antibacterial purposes.

In this work we loaded the TiO₂ nanofiber matrix with the well-known antibacterial Curcumin drug. We have generated an absorption spectrum of the desorbed Curcumin from the fiber matrix. It is found that the fibers with fewer diameters are having more amount of drug adsorption.

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