Enhanced Approach for Producing Zirconia Nanofiber

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Abstract: Electrospinning is a technique of producing nanofibers from polymer solution under the influence of electrostatic forces. It provides a relatively inexpensive method of creating a variety of nanofibers. In this work, the formation of zirconia nanofibers by electrospinning technique was investigated. Nano fibers of zirconia were prepared in three forms under different weight percentage as well as under optimized condition too. As a end result three different zirconia nanofibers were produced and characterized by Fourier transform infrared (FTIR) spectrometry, UV and SEM.

I. INTRODUCTION

ZrO_2 is a polymorphic material is also known as zirconia. It is a wide band-gap p-type semiconductor so it can be used in gas analyzers, sensors etc., Abdul-Majeed Azad *et al*, achieved fabrication of uniform 1-D nanofibers from alcoholic solution containing polyvinyl pyrrolidone (PVP) and aqueous precursors of yttrium and zirconium ions[1].similarly preparation of PVA by dissolving it in a distilled water and heating at 80^{o}c for 2hrs was reported. In this work a dense uniform fibres were collected in the substrate[3]. Defects free uniform fibres were obtained by varying applied voltage ranges between 12 to 16KV. The end product was nanohybrid mats, these mats contains highest quantity of nanoparticles which shows significant reduction of crystallinity [4].By adopting different synthesis conditions, formation of nanobars and hexagonal shaped nanodisc can also be done[5]. By using electrosppinning technique, uniform 1-D nanfibers from alcoholic solutions like PVP were achieved [6]. Similarly micromechanism studies and catalytic property of zirconia/PVP electrospun nanofibers were done under optimal conditions [7]. By calcinating and sintering PVA/zirconium acetate into metal oxide was produced [8]. Different dimensions of electrospun ZrP/PVA polymer nano composites were successfully produced by electrospinning techniques [9]. Electrospun zirconia nanofibers properties like polymer decomposition , crystallisation formation, phase transformation, surface morphologies were investigated [10].

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II. METHODOLOGY

Electrospinning is an essential and resourceful method to produce polymers by increasing the rate of electric field in charged polymer jet.

The resultant fibers ranges between 10µm to 10nm, which is typically 3 times lesser than that of the fiber obtained by conventional spinning method. A schematic diagram of electrospinning setup is shown in (Fig. 1.a). The major components of this method are (i) high voltage power supply (ii) syringe (iii) substrate (iv) rotating drum (v) DC motor. Firstly the syringe is loaded with melted polymer solution and it is connected to positive terminal of the high voltage power supply.

During this process a high voltage is used to form an electrically charged jet of polymer melt. In this technique, positive terminal of the electrode is connected to the melt and negative terminal is attached to the collector which is grounded. This indicates the charged particles by the electric field which are accumulated at the tip of the needle that support the solution's droplet held by its surface tension. This process begins when a high voltage is applied to the electrode, the electric charges moves into polymer solution. As a result of this, the induced charge within the solution causes instability of the polymer. Simultaneously, repulsion of charges produces an electrostatic force against the surface tension that allows the solution flows in the direction of electric field. By increasing the intensity of electric field , it makes the droplet to elongate, in order to form an inverted cone which is popularly known as Taylor cone. At this stage a fine elongated polymer strands emerge from Taylor cone , which are collected on the metallic substrate kept at an optimized distance

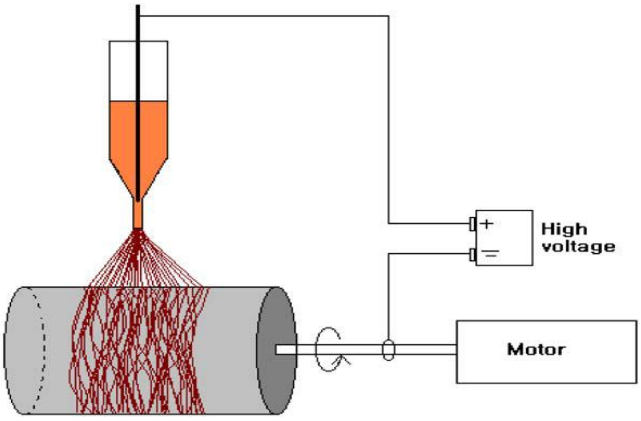

Fig. 1.a Schematic diagram of electrospinning setup

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III. FACTORS AFFECTING ELECTROSPINNING PROCESS

There are so many factors that affect the electrospinning process. They are

(i) applied voltage
(ii) polymer solution
(iii) distance between needle to substrate

Effect of Applied Voltage:

It is well known that the flow of current into the solution will cause Taylor cone at a critical voltage, to form ultrafine nanofibers.

This critical voltage differs from polymer to polymer. If the applied voltage is above the critical value it will result in beaded shape nano fibers, similarly it will reduce the size of the Taylor cone. Once the size of the Taylor has been reduced the solution gets accumulated at the needle tip. So that there will be droplets in the substrate instead of fibers.

Polymer Solution

To fabricate a uniform fibres, surface tension, surface charge , viscosity plays a major role. Surface tension tries to minimize specific surface area, by changing jets into spheres. On the other hand an excessive electrical charge tries to increase the surface area, which will help in making thinner jets. Increase in solution's viscosity will proportionally increases bead size. As net charge density increases the beads become smaller in size. Decreasing surface tension makes the beads disappear. So a thin, stable jet is necessary for a balance of viscosity, charge density, and surface tension.

Distance between Needle to Substrate:

The distance between the metallic needle tip and collector plays vital role in determining the morphology of an electrospun nanofiber. Similarly the applied electric field, the distance between the metallic needle tip and collector also varies with the polymer system. The nanofiber morphology could be easily affected by the distance because it depends on the deposition time, evaporation rate, and instability interval. Hence, a critical distance needs to be maintained to prepare uniform electrospun nanofibers.

Materials Used:

(i) polyvinyl alcohol(PVA)
(ii) zirconia
(iii) solvent(H_2O)

PVA:

Compared with any other known polymer, PVA comprises of superior features such as substantial tensile strength, more flexibility, oxygen barrier and aroma barrier. PVA is characterized by properties such as chemical resistance, water solubility, uniform flim formation and it is also biodegradable.

Zirconia:

Zirconia is an inorganic metal oxide that is mainly used in ceramic materials. Since it is a heavy metal, it supports properties such as hardness, low reactivity, high melting point and high optical density. Beside to that it has high mechanical stability which improves the property of scratch resistance and resistant to abrasion. Here we have used ZrO_2 in 3 different concentration, 20mg, 30mg and 40mg.

SOLVENT:

In the preparation of PVA , water is a solvent which plays an essential role. By adding PVA (5%)to de-ionized water at 60^oc and by stirring it for 3 hrs, 10% of PVA solution is got. Here the pH value of solvent is maintained less than 7 eventually, to get a good thin film forming and excellent chemical resistance.

Experimental Procedure

Initially, zirconia was taken in three different concentrations 20mg, 30mg, and 40mg. H_2O was used as a solvent, since it has high dielectric constant. Hence, the polymer PVA (5%) was dissolved in 5ml of water for 3 hours at 60°C. As a result, fine film of PVA, zirconia is obtained. Then zirconia was suspended in dissolved PVA for 24 hours. Before loading the solution in the syringe excess air should be removed, then it is set in the syringe pump. There is a knob in the bottom of the syringe pump which can also be adjusted to bring the liquid to the needle tip and the syringe is holded by the syringe holder. The power supply delivers a maximum of 20KV. The distance between the needle tip and the rotating drum was 20cms. The flow rate was varied to obtain stable jet. The flow rate was maintained at 0.5ml/hr. By applying high voltage, a taylor cone was formed on the needle tip and the nanofibers were collected on the rotating drum.

IV. RESULTS AND DISCUSSION

Optical Studies:

The optical study of solid concerns not only with the physical phenomena such as refraction, reflection, transmission, absorption, polarization and interference of light but also the interaction of photon energy with matter and the consequent changes in the electronic states. Absorption of light by different materials can induce various types of transitions such as band to band, between sub-bands, between impurity levels and bands, transitions of free carriers within a band and also resonance due to vibration state of lattice and impurities. These lead to the appearance of bands or absorption peaks in the absorption spectra.

The optical method provides a very simple way of finding the band gap as compared to the method using thermal excitation, which is less reliable. To determine the band gap of the thin film, the following procedure has to be adopted from the optical measurements. The absorbance (log (I_o / I), I_o- Intensity of the incident light, I – Intensity of the transmitted light) for each wavelength of light at normal incidence on the film is obtained using a ELICO SL 159 UV-VIS Spectrophotometer

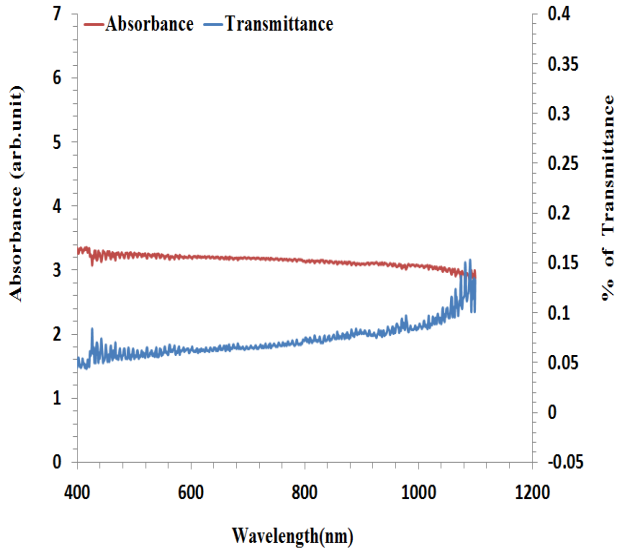

Fig. 1.b Optical absorbance and transmission of 20mg of zirconia/PVA sample

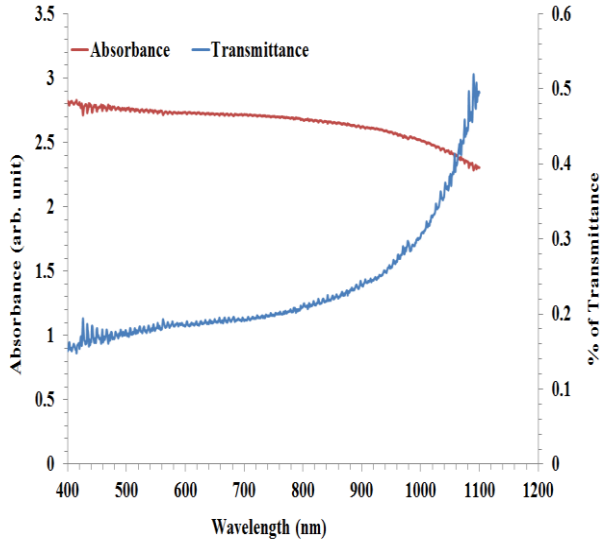

Fig. 1.c Optical absorbance and transmission of 30mg of zirconia/PVA sample

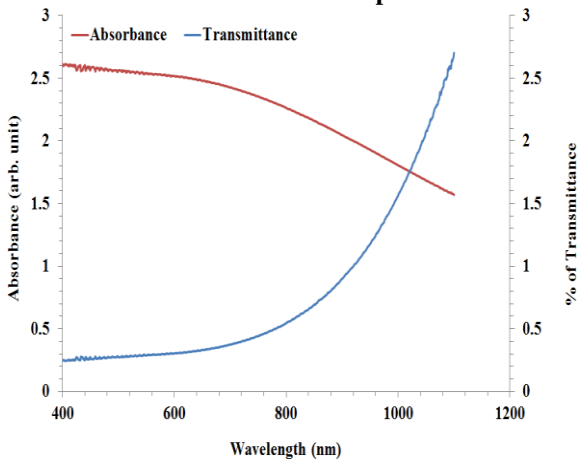

Fig. 1.d Optical absorbance and transmission of 40mg of zirconia/PVA sample

The fig 1.d. shows optical absorbance and transmittance of zirconia film prepared at optimized condition. In 3 different concentration of Zirconia the maximum transmittance occurs at 2.75% at 1100nm. The spectrum shows a sharp and prominent absorption band with maximum at around 330 nm which can arise due to the transition between valence band to conduction band. Apart from the strong absorption peak, a broad and weak peak centred at around 330 nm exists. As a result the maximum absorption occurs in the 20mg concentration of zirconia and maximum transparency is at 2.5%. Hence the material shows maximum transparency in 40mg of concentration of zirconia.

1. FT-IR Studies:

Fig.1.e. shows the FT-IR spectra for the PVA/zirconia composite fibres and for those calcined at different concentration. As observed in Figure, due to the concentration of 30mg of Zr in 5 wt% of PVA , the voltage was maintained at 20KV and the flow rate of the solution was 0.5ml/hr. The peaks at about 3303, 2939, 2911,1710,1435,1238, 918, 850, 608cm^{-1} corresponding to C-H; C-C; C-O;O-H; respectively , two new peaks around 1095 and 607 cm^{-1} assigned to ZrO_2.These results suggested that the presence of zirconia is there in all concentration.

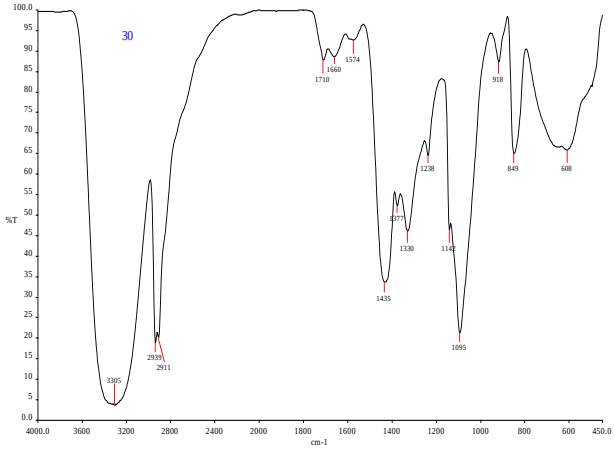

Fig. 1.e FT-IR spectra for the PVA/zirconia composite Fibres

2. SEM Results:

The SEM result shows electrospun zirconia fibers. Since there is no beads it states that the material is well suspended in the solvent. The average diameter of the fibers is of 1 µm.

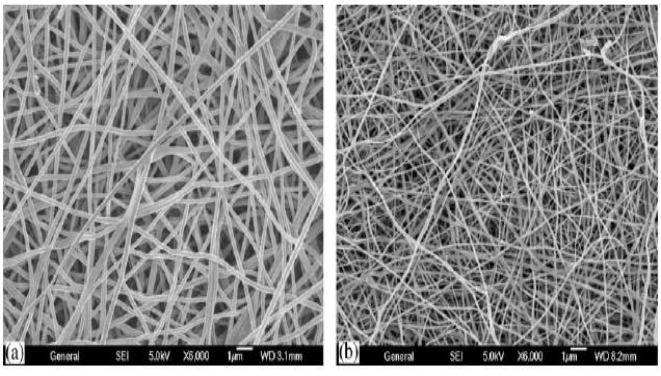

Fig. 1.f scanning electron microscope of PVA/zirconium fiber samples

V. CONCLUSION

Zirconia fine powder has been prepared by electrospinning method based on optimized conditions like high power supply of 20KV, flow rate of 0.5ml/hr, speed of the rotating drum, distance between the needle tip and the substrate. In this experiment it was carried out on three different concentration like 20, 30, 40mg in 5wt% of PVA. The optical studies shows the absorption and transmittance of three different samples. In this the maximum absorption peak was observed at around 330 nm which can arise due to the transition between valence band to conduction band. Hence the material shows maximum transparent in 40mg of concentration of zirconia.

As a result there was smooth absorption and less transparency was observed in the samples. The FTIR spectra shows the presence of zirconia. Two new peaks around 1095 and 607 cm^{-1} assigned to ZrO_2.These results suggested that the organic molecules could be removed completely from PVA/zirconia composite fibres. The surface morphology of zirconia flim prepared from the optimized condition and in three different concentration of zirconia. It shows that the material that it is deposited on the aluminium foil which shows well-defined fiber texture. These fibers are randomly distributed. Most of the fibers are dense. The average diameter of these fibers is in the range of 0.8 to 1 μm and the length could be reached to several millimetres as shown in the fig. Since there is no beads in the morphology it shows that the material is well suspended in the solvent.

VI. FUTURE WORK

As it is well known that zirconia is a good dielectric material in future it can be used in transistor to achieve maximum efficiency. In addition to that it is also possible to eliminate the instability and phase transformation, by adding stabilizing material like magnesia, calcium oxide or yittrium to develop different zirconia composite.

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