

# Influence of Variables on Synthesis of NiO/Al<sub>2</sub>O<sub>3</sub>/B<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> Heterostructured Nanofibers via Sol-Gel Route and Electrospun Method

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**Abstract**—Nanofibers of PVA /nickel nitrate/silica/alumina izopropoxide/boric acid composite were prepared by using sol-gel processing and electrospinning technique. By high temperature calcinations of the above precursor fibres, nanofibers of NiO/Al<sub>2</sub>O<sub>3</sub>/B<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> composite with diameters about 500 nm could be successfully obtained. The fibres were characterized by XRD and SEM analyses.

**Keywords**—Nanofibers, ceramics composite, sol-gel processing, electrospinning.

## I. INTRODUCTION

NANOMATERIALS have become a research priority as biotechnology, defense and semiconductor industries in particular, are interested in potential applications of nanotechnology. Specifically, a substantial amount of research on nano-scale fibers is being conducted to meet the demands of their prospective application areas such as tissue engineering, membranes, nano-resonators, micro-air vehicles and hydrophobic thin films. In recent years, nano structural materials such as nanorods, nanowires and the nanofibers have been actively studied due to both their scientific use and potential applications in nanodevices. In particular, the dispersion of metal nanoparticles in an inorganic matrix has aroused great interest. Nickel oxides are well-studied materials due to their high activity, low cost, and abundant element, nickel-based catalysts have been extensively employed and investigated for catalysis, chemical and energy applications. [1]-[4].

Electrospinning, a spinning technique, is a unique approach using a rapid forces to produce fine fibers from polymer solutions or melts and the fibers thus produced has a thin inner diameter (from nano meter to micrometer) and a larger surface area than those obtained from conventional spinning processes. Furthermore, a DC voltage in the range of several tens of kV is necessary to generate the successful defence of [5].

The sol-gel method has been compounded with the electrospinning to produce very fine polymer/ceramic nanofibers. The chemical and physical features of the materials obtained by these methods (e.g., surface areas, particle sizes and mechanical properties) can be changed with the temperature, operating conditions, and the used precursor, which means that control, is provided over the microstructural properties of these materials thanks to this technique. The essential feature of this method is to give a desirable particle size and distribution at high yield and low preparation cost [6]. This study reports an easily applicable and

inexpensive approach of the fabrication of the nickel alumina borosilicate composite nanofibers using the sol-gel processing and the electrospinning technique. The PVA /nickel nitrate/aluminium izopropoxide /boric acid/TEOS (Tetraethyl orthosilicate) composite was used as precursor through calcination treatments.

## II. EXPERIMENTAL PROCEDURE

In this study, mixture which was prepared by using three different PVA solutions was subjected to electrospinning process. Compositions of solutions A, B and C were given in Table I.

TABLE I  
COMPOSITIONS OF ELECTROSPINNING SOLUTIONS

<b>Sol.-A</b>	TEOS:EtOH: H <sub>2</sub> O:HCl	1:10:2:0.01 (molar)
<b>Sol.-B</b>	Ni(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O:EtOH:HCl	1:10:8 (molar)
	(Ni(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O:EtOH:HCl):PVA	1:1 (by weight)
	H <sub>3</sub> BO <sub>3</sub> :EtOH:HCl	1:10:8 (molar)
	(H <sub>3</sub> BO <sub>3</sub> :EtOH:HCl):PVA	1:1 (by weight)
<b>Sol.-C</b>	Al(C <sub>3</sub> H <sub>7</sub> O) <sub>3</sub> :i-PrOH:HCl	0,1:2:0,8 (molar)
	(Al(C <sub>3</sub> H <sub>7</sub> O) <sub>3</sub> :i-PrOH:HCl):PVA	1:1 (by weight)

**Solution-A:** Tetraethyl orthosilicate (TEOS) and absolute ethanol (EtOH) were mixed in a beaker, then a certain amount of H<sub>2</sub>O/HCl solution was added slowly into TEOS/EtOH solution and this mixture was stirred. Solution-A was prepared about an hour later, when partial hydrolysis of TEOS occurred.

**Solution-B:** Nickel nitrate (Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O) was mixed with ethanol and HCl in a beaker, then a certain amount of PVA was slowly added to this mixture while stirring continues. Similarly, Boric acid (H<sub>3</sub>BO<sub>3</sub>) was mixed with ethanol and HCl in a beaker, and then a certain amount of PVA was slowly added to this mixture while stirring continues. Later; the solution containing nickel, was added slowly to the solution containing boric acid while stirring continues.

**Solution-C:** Certain amount of HCl, isopropyl alcohol and PVA were added onto aluminium isopropoxide (Al (C<sub>3</sub>H<sub>7</sub>O)<sub>3</sub>) respectively and this mixture was stirred until a clear solution was obtained.

**PVA Solution:** PVA which was in form of powder was stirred for an hour in distilled water at 80°C and then continued to

stirring at room temperature for 24 hours by the way PVA solution was prepared.

Solution B and solution C which were prepared as mentioned above were added slowly to solution A while mixture highly stirring and continued to stirring.

The distance between the tip of the capillary and metal collector was selected 10 cm. Electrospinning of the solution was performed at three applied voltages: 7, 10 and 15 kV. Solutions in different concentrations ranging between 7% PVA, 10% PVA and 12% PVA (w/w- weight - by weight basis) were prepared. Then, the formed fibres were dried initially at 110°C for 2 hours and calcined at a heating rate of 2°C/minute in air at 400 and 750°C temperatures, respectively. It remained at there quired temperature for 2 hours to obtain the NiO/Al<sub>2</sub>O<sub>3</sub>/B<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> nano fibres.

### III. RESULTS AND DISCUSSION

#### A. X-Ray Diffraction (XRD)

Fig. 1 shows the XRD patterns of nanofibres calcinated at 750°C. As shown in the figure, all the diffraction peaks were extremely similar to those of the nickel alumina borosilicate phase. In unison with Guan et al., the crystalline region of all of the Al<sub>2</sub>O<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and also NiO was observed [7]. The XRD patterns of the resulting composite nanofibers were found to be in accordance with the crystal structure and the literature value.

#### B. The Effect of Applied Voltage

In the electrospinning process a crucial element is the applied voltage to the solution. Only after attainment of threshold voltage, fiber formation occurs; this induces the necessary charges on the solution along with electric field and initiates the electrospinning process. It has been already proved experimentally that the shape of the initiating drop changes with spinning conditions (voltage, viscosity, and feed rate). There is a little dispute about the behavior of applied voltage in the electrospinning process. The changes in average fiber diameters with variations in applied voltage values were obtained by image analysis and are summarized in Table II.

As shown in Figs. 2 and 3, PVA concentration (10%), feed flow rate (3 µl/h) and the needle tip distance between the collectors (10 cm) is kept constant, the electric voltage applied to the needle is reduced with increased nanofiber diameter [8].

TABLE II  
AVERAGE FIBER DIAMETER CHANGES WITH ELECTRIC POTENTIAL

Electric Potential	Average Fiber Diameter (nm)
7 kV	945

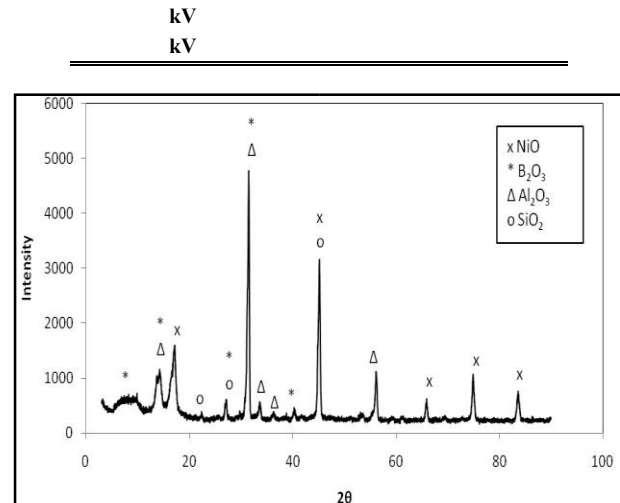
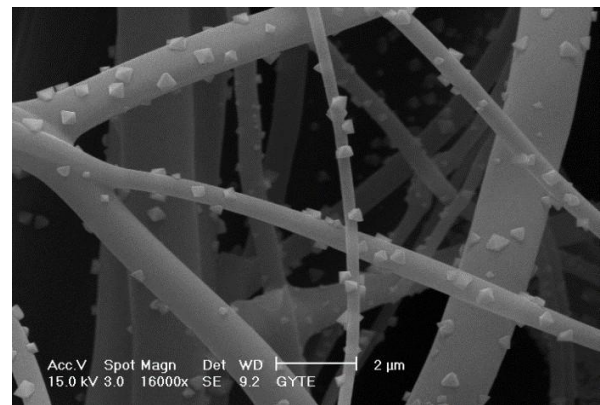
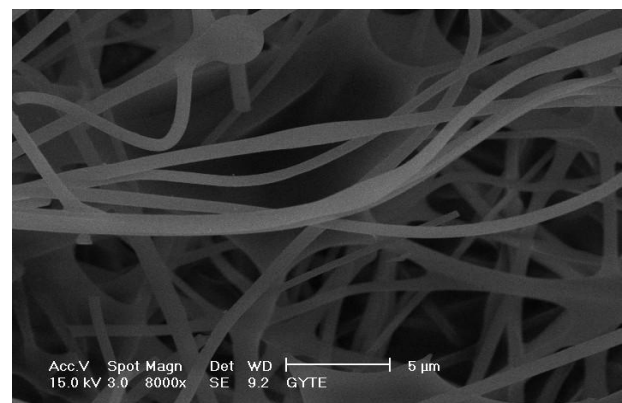


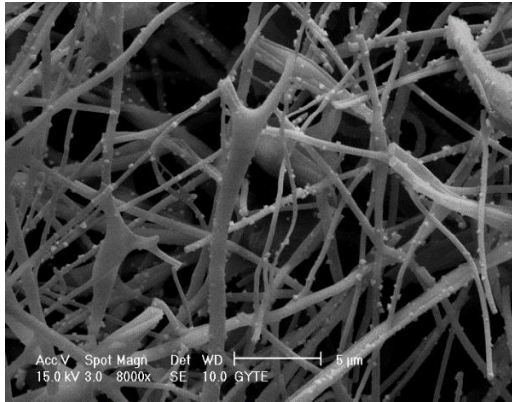
Fig. 1 XRD patterns of the NiO/Al<sub>2</sub>O<sub>3</sub>/B<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> nanofibers calcined at 750°C



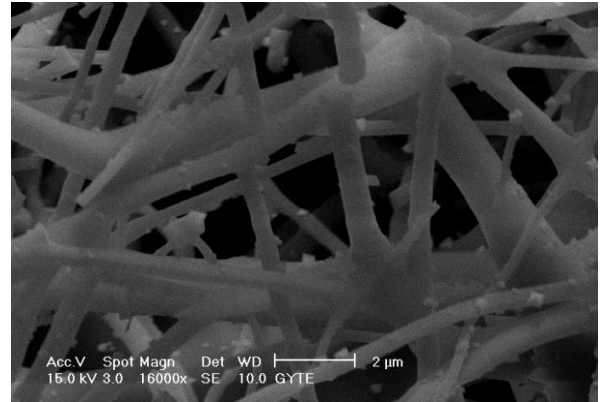
(a)



(b)



(c)



(a)

Fig. 2 Average fiber diameter changes with electric potential: (a) 7 kV, (b) 10 kV, (c) 15 kV

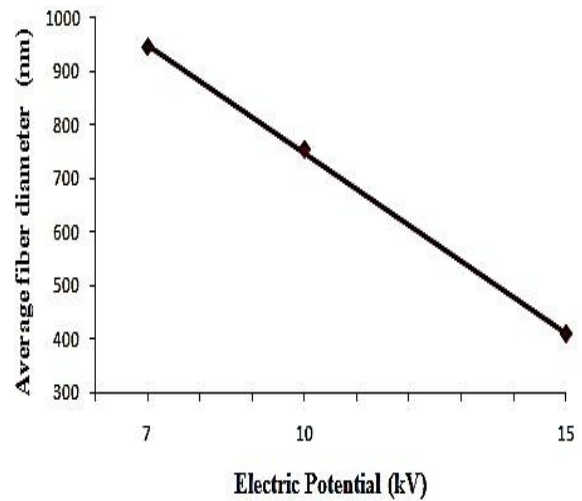
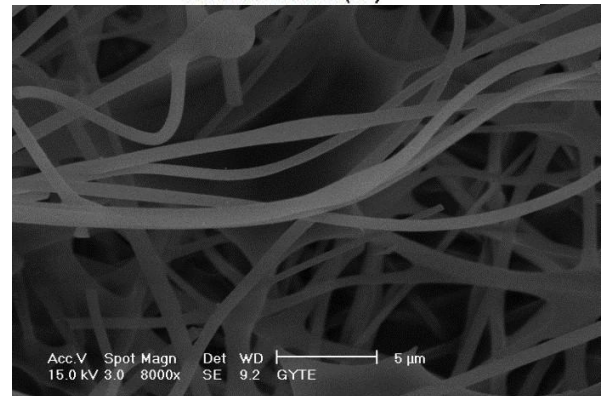


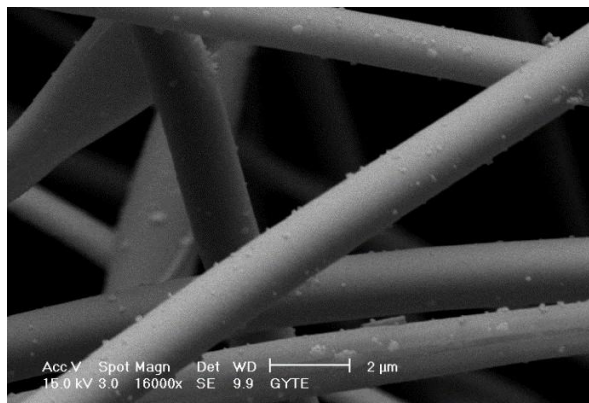
Fig. 3 Electrical potential (kV) effect of nanofiber diameter

TABLE III  
AVERAGE FIBER DIAMETER CHANGES WITH CONCENTRATION

Concentration (%)	Average Fiber Diameter (nm)
7 PVA	
10 PVA	
12 PVA	



(b)



(c)

Fig. 4 Average fiber diameter changes with concentration (a) 7 % PVA (b) 10 % PVA, (c) 12 % PVA

### C. The Effect of Concentration

In the electrospinning process, for fiber formation to occur, a minimum solution concentration is required. It has been found that at low solution concentration, a mixture of beads and fibers is obtained and as the solution concentration increases the shape of the beads changes from spherical to spindle-like and finally uniform fibers with increased diameters are formed because of the higher viscosity resistance. The changes in average fiber diameters with variations in concentration values were obtained by image analysis and are summarized in Table III.

As it is seen from Figs. 4 and 5, the electric potential applied to the needle (10 kV), the supply flow rate (3  $\mu\text{l/h}$ ) and the distance between the needle tip and the collector (10 cm) is kept constant, increased viscosity with increasing concentration, and the fiber diameter was thicker [9].

Fig. 5 Concentration (% PVA) effect of nanofiber diameter

## IV. CONCLUSION

In conclusion, nanofibres of the nickel alumina borosilicate composite have been successfully prepared using the sol-gel processing and the electrospinning techniques. A sol-gel recipe that allows the formation of a homogeneous four component alkoxide solution and provides a control over solution viscosity for electrospinning process was developed. This situation was thought to be due to the complete removal of organic molecules and the development of the nickel alumina borosilicate composite fibers. The effect of the spinning distance was more pronounced at higher applied voltages. Increasing the applied voltage increases the surface charge of the jet and helps to reduce the frequency of occurrence of beads. Viscosity increased with increasing concentration and consequently thickened fiber diameter but there was a more uniform and without bead structure.

Solution and processing parameters such as viscosity, molecular weight, concentration of the polymer, applied voltage, tip to collector distance, conductivity, etc. significantly affect the

fiber morphology and by manipulation of these parameters one can get desired properties for specific application.

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