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The Effect of the Distance between Needle Top-Collector on Production of Nickel Borate Nano Fiber by using Sol-Gel Method

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Abstract- In this work, applicability of the use of sol-gel and electro spinning techniques in preparation of nickel borate ceramic nanofibres was demonstrated. Electro spinning of the precursor solutions was ensured by the adjustment of viscosity of the solutions with polymeric additions. Electro spinning of these solutions into nanofibres composites was followed by the calcination of the electro spun fibers to obtain pure ceramic fibers. The nickel borate Nano fibers was produced by calcinations at the certain temperature 750 ^{o}C of the nanofibres obtained with this method.

Keywords- *Composites, Ceramics, Nanostructures, Sol-gel chemistry, electrospinning process*

I. INTRODUCTION

Composite nanofibres can be formed in many different deposition techniques. A sol-gel recipe that allows the formation of a homogeneous four component alkoxide solution and provides a control over solution viscosity for electro spinning process was developed. In addition to, electro spinning is the most reliable and commonly used techniques to obtain desired fibre properties. Nano materials have become a research priority as biotechnology, defense and semiconductor industries in particular, are interested in potential applications of nanotechnology. In particular, the dispersion of metal Nano particles 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. The electro spun nanofibres are able to form a highly porous mesh and their large surface-to-volume ratio improves performance for numerous applications [1-3].

New identification techniques using sol gel technology has started to progress quite a number of scientific studies. 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 [4]. This study reports an easily applicable and inexpensive approach of the fabrication of the nickel borate composite nanofibres using the sol-gel processing and the electro spinning technique. The product obtained in this way was characterized by FT-IR (Fourier transform-infrared spectroscopy), SEM (Scanning electron microscopy), and XRD (X-ray diffractometry).

II. MATERIALS AND METHODS

The sol-gel solutions used in the electro spinning were composed of nickel nitrate ($Ni(NO_3)_2.6H_2O$, Sigma-Aldrich, >98%), tetraethyl orthosilicate (TEOS, $Si(C_2H_5O)_4$, Sigma-Aldrich, >98%), aluminum isopropoxide ($Al(OC_3H_7)_3$, Sigma-Aldrich, >97%), boric acid (H_3BO_3, Etibank A.Ş), polyvinyl alcohol (PVA Mw = 80.000 g/mol, Sigma-Aldrich), hydrochloric acid, absolute ethanol (EtOH), isopropyl alcohol (i-PrOH) and deionized water.

PVA aqueous solution (10wt%) was first prepared by dissolving 10 g PVA powder in 100 ml of distilled water while stirring for 2 hours at 80^{o}C, then the solution was cooled to room temperature and stirring was maintained for 12 hours. A silica gel solution was prepared by mixing 10 ml aluminum isopropoxide ($Al(OC_3H_7)_3$) 10 ml of tetraethyl ortosilicate (TEOS), 10 ml of ethanol, 10 ml of water, 10 ml isopropyl alcohol and 2 ml of concentrated HCl together. The mixed solution was maintained in a 40^{o}C water bath with vigorous magnetic stirring for 2 hours. In another container, 10 ml of boric acid and 20 ml of PVA was mixed with 10 ml of nickel nitrate. Next, the above solutions were separately added into the prepared 30 ml PVA aqueous solution under vigorous stirring at a slow speed. The pH of medium is below 2 to protect of peptization during the forming of the sol-gel. The resulting solution was placed in a 10 ml plastic capillary-tipped syringe. Fibers on a metal collector which is the aluminum foil were collected in the electro spinning apparatus under these conditions that are 10 kV, capillary tip to metal collector distance of 10 cm, 10% PVA solutions and 3 ml/h flow rate of solution. Thus, the formed fibres were dried initially at 110^{o}C for 2 hours and then, calcined at a heating rate of 2^{o}C/minute in air at 400, 750^{o}C, and 1000^{o}C temperatures, respectively. It remained at the required temperature for 2 hours to obtain the nickel borate composite nanofibres.

III. RESULTS AND DISCUSSION

A. FT-IR Spectra

According to Fig. 1a, the peaks are shown, especially Si-O-Si at around 1091 cm^{-1}. When calcinations were at 750°C and 1000°C (Fig. 1b-c), all of the peaks fitting to the organic groups of PVA and other components faded away and a new peak at 467 cm^{-1} is appointed to the Ni-O bond [5]. Formation of the anticipated B-O-Si linkage is confirmed by (FT-IR). The FT-IR spectrum of nanofibres indicates the presence of the B-O bonds at around 1430 cm^{-1}. In particular, this distinct band assigns the asymmetric stretching of the BO_3 unit [6]. In addition, in the all of the FT-IR spectra, the peaks at around 800 cm^{-1} are assigned Al-O-Si (bending). In addition, when the composite is calcinated, a peak at 1331 cm^{-1} assigned to Si-OEt or ethanol normally disappears [7].

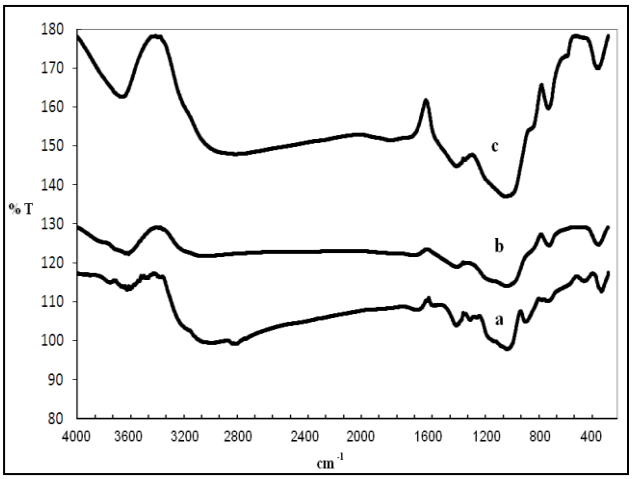


Figure 1. FT-IR spectra for (a) before applied the thermal treatment nickel borate composite fibres; (b) calcined at 750 °C; (c) calcined at 1000 °C

B. X-Ray Diffraction (XRD)

Figure 2 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 borate nano fibers [5].

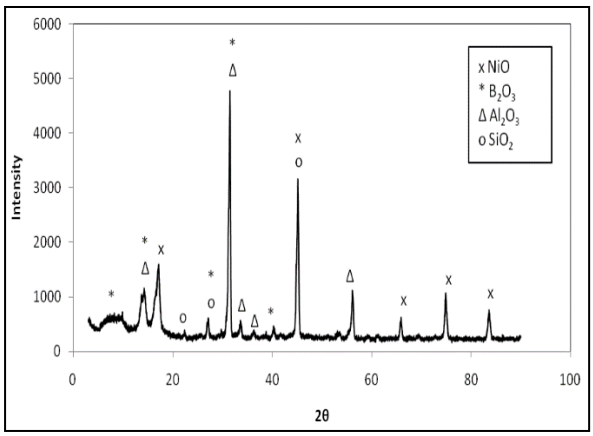


Figure 2. XRD patterns of the nickel borate nanofibers calcined at 750 °C

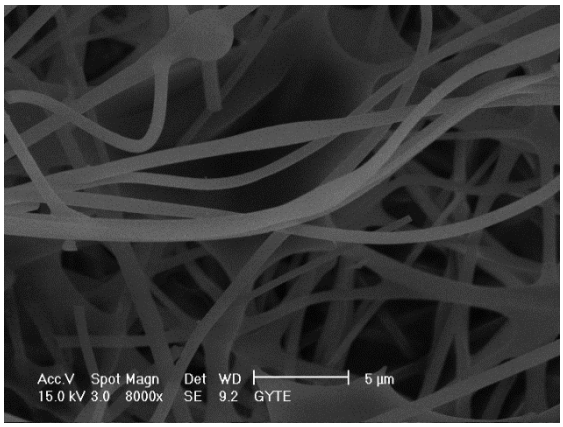
C. The effect of the distance between needle top-collector

The distance between the tip and the collector has been examined as another approach to control the fiber diameters and morphology. It has been found that a minimum distance is required to give the fibers sufficient time to dry before reaching the collector, otherwise with distances that are either too close or too far, beads have been observed. The changes in average fiber diameters with variations in tip to collector distance values were obtained by image analysis and are summarized in Tables 1 and Figure 3.

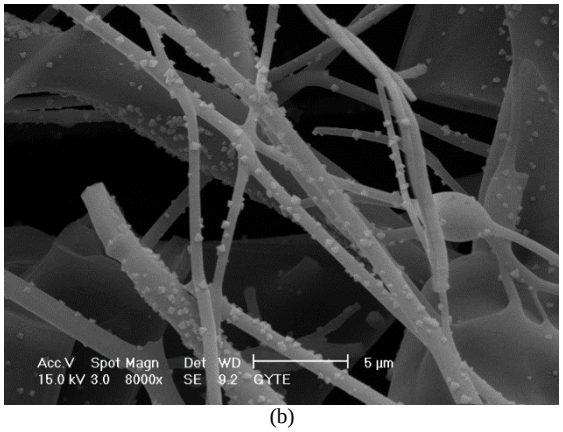
TABLE I. WITH A CHANGE OF THE DISTANCE BETWEEN THE TIP OF THE NEEDLE WITH THE COLLECTOR, THE VARIATION OF THE AVERAGE DIAMETER OF THE FIBERS

the distance between the tip of the needle with the collector (cm)	Average fiber diameter (nm)
10	754
15	680
20	560

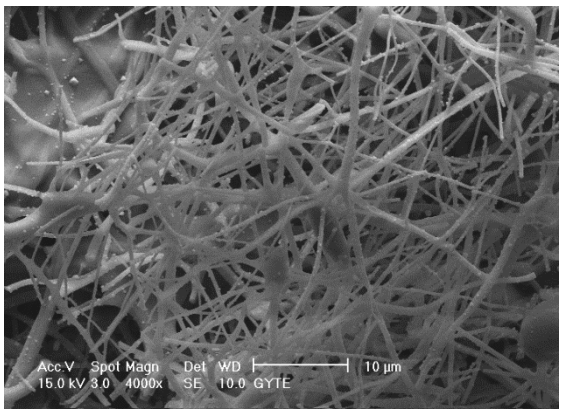
Figures 3 and Figure 4, as is recognizable, the electric potential applied to the needle (10 kV), the supply flow rate (3 µl/h) and the PVA concentration (%10) is kept constant, increased distance between the capillary needle tip to metal collector with increasing and the fiber diameter was smaller [8]. Solution and processing parameter as the distance between the tip and the collector significantly affect the fiber morphology and by manipulation [9].



(a)



(b)



(c)

Figure 3. With a change of the distance between the tip of the needle with the collector, the variation of the average diameter of the fibers. (PVA concentration %10, 10 kV Electrical Potential and 3 µl/h flow volume of solution): *Variables: 10 cm (a) 15 cm (b) 20 cm (c)

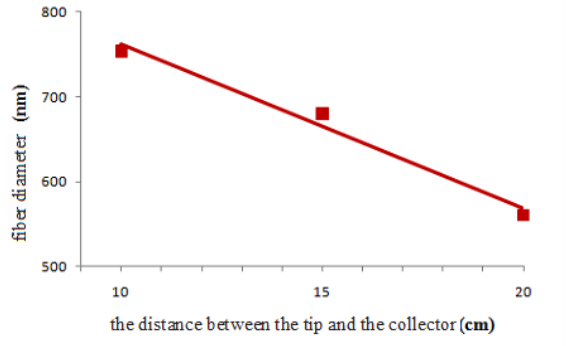


Figure 4. The effect of variation of fiber diameter the distance between the tip and the collector (PVA concentration %10, Electrical Potential 10 kV and flow volume of solution3 µl/h)

IV. CONCLUSIONS

The distance between the tip and the collector has been examined as another approach to control the fiber diameters and morphology. In conclusion, nanofibres of the nickel borate composite have been successfully prepared using the sol–gel processing and the electro spinning techniques. This situation was thought to be due to the complete removal of organic molecules and the development of the nickel borate composite fibers. The effect of the spinning distances was more pronounced at higher applied voltages. Borates are structurally analogous to silicates except for the modes of coordination. Silicates are coordinated to oxygen in a tetrahedral geometry while borates can coordinate to oxygen both in triangular and tetrahedral geometry. Nevertheless, it is still not easy to foretell which coordination of boron will dominate in a structure at environmental conditions. The side of the best of our works there is no presented on nickel borate fibers although various morphologies of metal borates have been investigated.

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