



Preparation and Characterization of PbO.ZrO₂.TiO₂ and SrO.ZrO₂.TiO₂ Nanofibers by Electrospinning Method

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Abstract

Lead zirconate titanate (PZT)-based ferroelectric ceramic materials are piezoelectric materials of commercial importance. Strontium zirconate titanate (SZT) perovskite materials also have a wide application area. PZ and SZT starting solutions can be prepared using a variety of suitable methods. Among these methods, the sol-gel process has attracted special attention due to the low temperature requirement, homogeneity and the ability to obtain the appropriate particle size. The electrospinning method is the most effective and widely used method in nanofiber production. Electro-spinning is a method that enables the production of solid and hollow nanofibers in long lengths, homogeneous diameters and various compositions. When the electro-spinning technology is combined with the sol-gel method, continuous nanofibers, nanotubes and filled nanofibers are produced from polymer or ceramic solution under a certain electric field.

In this study, lead zirconate titanate (PZT) and strontium zirconate titanate (SZT) solutions containing Sr and Pb were prepared by sol-gel method. Solutions were prepared using metal salts and alkoxides as the precursor materials. The solutions obtained were fed at flow rates of 1.6, 1.8, 2.0 mL / hour and PZT and SZT nanofibers were formed by electrospinning method at 20 kV voltage rating. Different parameters of the electrospinning device were examined and the SZT and PZT nanofibers were produced with optimum properties. Crystal structure, characterization of morphological surface and chemical properties of the produced nanofibers were done with XRD, SEM and EDX, respectively. XRD results showed that the perovskite structure of PZT and SZT was not damaged during Sol-gel and electrospinning processes. A small amount of beady structure was observed in SEM analysis of PZT and SZT nanofibers. According to EDX analysis of nanofibers, it was observed that the PZT and SZT structure was also formed.

Keywords: PZT, SZT, Sol-Gel Method, Electrospinning Device.

PbO.ZrO₂.TiO₂ ve SrO.ZrO₂.TiO₂ Nanofiberlerin Elektrospinning Yöntemi ile Hazırlanması ve Karakterizasyonu

Öz

Kurşun zirkonat titanat (PZT) bazlı ferroelektrik seramik malzemeler ticari öneme sahip piezoelektrik malzemelerdir. Stronsiyum zirkonat titanat (SZT) perovskit malzemeleri de geniş bir uygulama alanına sahiptir. PZT ve SZT başlangıç çözeltileri uygun olan çeşitli yöntemler kullanılarak hazırlanabilir. Bu yöntemler arasında sol-jel işlemi, düşük sıcaklık gereksinimi, homojenlik ve uygun partikül boyutunun elde edilebilmesi nedeniyle özel ilgi görmüştür. Elektroçirme yöntemi nanofiber üretiminde etkin ve en yaygın kullanılan yöntemdir. Elektroçirme, katı ve boşluklu içyapılı, uzun boylarda, homojen çapta ve çeşitli bileşimlerde nanofiber üretimi sağlayan bir yöntemdir. Elektroçirme teknolojisi sol-jel yöntemi ile birleştirildiğinde, polimer veya seramik çözeltiden belirli bir elektrik alan altında sürekli nanofiber, nanotüp ve dolgulu nanofiber üretimi gerçekleştirilmektedir.

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Bu çalışmada, Sr ve Pb içeren kurşun zirkonat titanat (PZT) ve stronsiyum zirkonat titanat (SZT) çözeltileri sol-jel yöntemi ile hazırlanmıştır. Çözeltiler, öncü malzemeler olarak metal tuzları ve alkoksitler kullanılarak hazırlanmıştır. Elde edilmiş olan Çözeltiler, 1.6, 1.8, 2.0 mL / saat akış hızlarında beslenmiştir ve 20 kV voltaj değerinde PZT ve SZT nanofiberleri elektrospinning yöntemi ile oluşturulmuştur. Elektroğirme cihazının farklı parametreleri incelenmiş ve SZT ve PZT nanofiberleri optimum özelliklerde üretilmiştir. Üretilen nanofiberlerin kristal yapısı, morfolojik yüzey karakterizasyonu ve kimyasal özellikleri sırasıyla XRD, SEM ve EDX ile yapılmıştır. XRD sonuçları Sol-jel ve elektrospinning işlemleri sırasında PZT ve SZT'nin perovskit yapısının bozulmadığını göstermiştir. PZT ve SZT nanofiberlerin SEM analizlerinde az miktarda boncuksu yapı gözlemlenmiştir. Nanofiberlerin EDX analizine göre PZT ve SZT yapısının da oluştuğu görülmüştür.

Anahtar Kelimeler: PZT, SZT, Sol-Jel Metodu, Elektroğirme Cihazı

1. Introduction

Development and production of energy generators operating with high efficiency have been needed due to the increasing need for energy in recent years. For this purpose, scientists have carried out many studies on the development of materials with piezoelectric, ferroelectric and pyroelectric properties due to their many application areas [1-3].

When pressure is applied on materials with piezoelectric crystal properties, electricity can be easily obtained by the applied pressure effect. This effect is directly related to the change in polarization density inside the material. If the piezoelectric material is not short-circuited, the applied pressure creates a voltage in the material. Because of this reason, piezoelectric ceramics are enormous materials that can convert mechanical energy into electrical energy. Piezoelectric ceramics are used in the computer industry (inkjet printers, disc drives), automotive industry (alarms, airbag sensors, fuel jets, air flow sensors, parking sensors, seat belt alerts), medical and military systems. It also forms the basis of many scientific techniques (such as STM, AFM, MTA, SNOM, which are scanning probe microscopes) in terms of atomic dissolution [4-6].

While describing the electrical behavior of the material, it is necessary to mention the characteristics of Pyroelectric materials. Pyroelectric materials have the ability to generate a potential difference between their ends when heated or cooled. Even a small temperature change at hundreds of degrees can be detected with pyroelectric materials. For this reason, pyroelectric crystals are commonly used in under-red detectors [7-9].

Ferroelectric materials have permanent polarization by themselves without being exposed to an external electric field. This polarization can be directed by the electric field effect. Ferroelectric materials are used in the production of capacitors with adjustable load storage capacity. These capacitors consist of a ferroelectric layer placed between a pair of electrodes. Ferroelectric materials must be symmetrical in order to show piezoelectric and pyroelectric properties. The combination of piezoelectric, pyroelectric and memory features makes ferroelectric capacitors very useful for sensor applications [10-12].

Lead Zirconate Titanate ($\text{Pb}(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3$ or PZT) is an excellent ferroelectric ceramic material that has perovskite structure,. On the other hand PZT and Strontium zirconate titanate ($\text{Sz}(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3$ or SZT) nanofibers have recently been widely developed by scientists, especially because of their ferroelectric, piezoelectric and pyroelectric properties, efficiency of energy conversion, large remnant polarization and because

they have many application areas [13-17]. The most widely used of these are sensors, actuators, ferroelectric memory devices, nano-generators, micro-electromechanical systems (MEMS) and converters [18-20]. On the other hand SZT ceramics have become a research subject in integrated microelectronics, microwave devices and electronic ceramics industry because of its high dielectric permeability, adjustability, low microwave loss, high breaking strength and low leakage current density [21, 22].

The electrospinning method is a simple, cost-effective and versatile process for the production of advanced fiber with varying diameters in micrometer and nanometer sizes in various fields [23-26]. Nanofibers are formed from solution or molten polymeric fluid using a high voltage electrical field.

For the polymer to be transported as fiber, it must be in liquid form or in the form of a polymer solution. The experimental setup required for the electrospinning method basically consists of three important parts as shown in Figure 1.1.

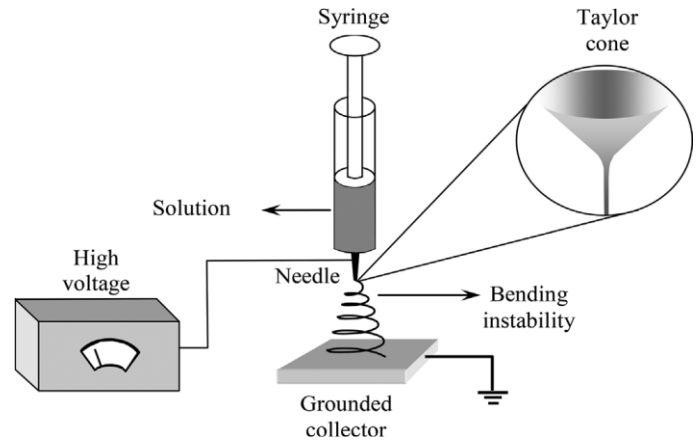


Fig. 1.1 Electrospinning experimental set-up [27].

1. High voltage power supply,
2. Feeding unit (syringe, metal needle, etc.),
3. Grounded collector (conductor plate, rotating cylinder, etc.).

The polymer and solvent are mixed at room temperature and a homogeneous polymer solution is prepared by sol-gel method. Then this solution is injected into the syringe. It is sent from the syringe to the metal needle at a constant speed using a pump. The high voltage source is turned on when the droplet takes the form of a cone at the tip of the metal needle. The polymer droplet is electrified and the induced droplet is evenly distributed

over the collector substrate surface. Many scientists used this method for the production of nanofibers.

In this study, PZT and SZT nanofibers containing Pb and Sr were prepared by sol-gel method using the electrospinning method. Solutions were prepared using metal salts and alkoxides as the precursor materials. Different parameters of the electrospinning device were examined and the SZT and PZT nanofibers were produced with optimum properties. Crystal structure, characterization of morphological surface and chemical properties of the produced nanofibers were done with XRD, SEM and EDX, respectively.

2. Material and Method

Lead zirconate titanate (PZT) and strontium zirconate titanate (SZT) nanofibers are obtained by electrospinning of sol-gel based solutions. PZT solution was prepared from lead Acetate, zirconium Oxychloride, titanium isopropoxide, and SZT solution was prepared from strontium nitrate, zirconium Oxychloride, titanium isopropoxide.

To prepare the solutions of nanofiber (SrO.ZrO₂.TiO₂) and nanofiber (PbO.ZrO₂.TiO₂), strontium nitrate, zirconium oxychloride, titanium isopropoxide, and lead acetate were chosen as precursors. Chemicals used in the experimental study are shown in Table 2.1.

Table 2.1. Chemicals Used in Experimental Study

Compound Name	Formulas	Properties
Lead acetate	Pb (CH ₃ COO) ₂ .3H ₂ O	Sigma, 99,9 % Mw= 379.34 g/mol
Strontium nitrate	Sr(NO ₃) ₂	Sigma, 99,995%, Mw=211.6 g/mol
Zirconium oxychloride octahydrate	ZrOCl ₂ .8H ₂ O	Sigma, 99.5%, Mw= 322,28 g/mol
Titanium isopropoxide	Ti{OCH(CH ₃) ₂ } ₄	Sigma, 99.5%, Mw=284.22 g/mol
Polyvinylpyrrolidone	(C ₆ H ₉ NO) _n	Sigma, M _w ~1,300,000
Absolute ethanol	CH ₃ CH ₂ OH	Sigma, 99.8%, Mw=46 g/mol

2.1. Preparation of Nanofiber Solutions (PbO.ZrO₂.TiO₂ and SrO.ZrO₂.TiO₂)

For the first step in the production of solutions of PZT nanofibers by sol-gel method, 1% of the molecular weight of lead, zirconium and titanium were taken. 5 grams of pure water was added to the Lead Acetate solution, and 3 grams of pure water to each of Zirconium Oxychloride and Titanium Alcohol solutions.

P: 379.34 g/mol → **1% 3.79 g/mol + 5 g pure water**
Z: 322.28 g/mol → **1% 1.67 g/mol + 3 g pure water**
T: 284.25 g/mol → **1% 1.36 g/mol + 3 g pure water**

And then, production of solutions of SZT nanofibers by sol-gel method, 1% of the molecular weight of strontium, zirconium and titanium were taken. 5 grams of pure water was added to the Strontium nitrate solution, and 3 grams of pure water to each of Zirconium Oxychloride and Titanium Alcohol solutions.

S: 211.63 g/mol → **1% 2.11 g/mol + 5 g pure water**
Z: 322.28 g/mol → **1% 1.67 g/mol + 3 g pure water**
T: 284.25 g/mol → **1% 1.36 g/mol + 3 g pure water**

The mixture was obtained by adding the two solutions into Titanium Alcohol solution. This prepared mixture solution was mixed in a magnetic stirrer at 250 rpm for 4 hours. After this process, 2g of PVP / 10ml of Ethanol were added slowly onto this solution and stirring was continued for 1 more hour. After the mixing process, the top of the beaker was covered with a watch glass and left to age for 1 day. PZT nanofibers were produced by the electrospinning method. The aged solution was drawn into a 10 ml 22 gauge plastic syringe and placed in the syringe pump. The solution was fed at 1.6, 1.8, 2.0 mL / hr flow rates and fibers were formed on aluminum foil at 20 kV voltage. PZT and SZT nanofibers formed on aluminum foil were heat treated at 1100 °C for 1 hour. One of the biggest problems encountered in nanofiber production is the loss of fibers after heat treatment. Three samples, A1, A2 and A3, were separated from the PZT solution. The same experimental steps were repeated to obtain SZT nanofibers. Thus B1, B2 and B3 samples were obtained. Table 2.2 showed that voltage and magnification values of PZT and SZT nanofibers.

Table 2.2. Voltage and magnification values of PZT and SZT nanofibers

Samples	Fibers	Voltage (kV)	Magnification (Kx)
A1	PZT	20	5.00
A2	PZT	20	10.00
A3	PZT	20	10.00
B1	SZT	20	5.00
B2	SZT	20	10.00
B3	SZT	20	10.00

Then the surface morphologies and chemical properties of group A and group B nanofibers have been analyzed in AKU Technology Application and Research Center by scanning electron microscope (SEM) and electron X-ray (EDX) measurements, respectively. The experimental steps are shown in Fig 2.1 and 2.2.

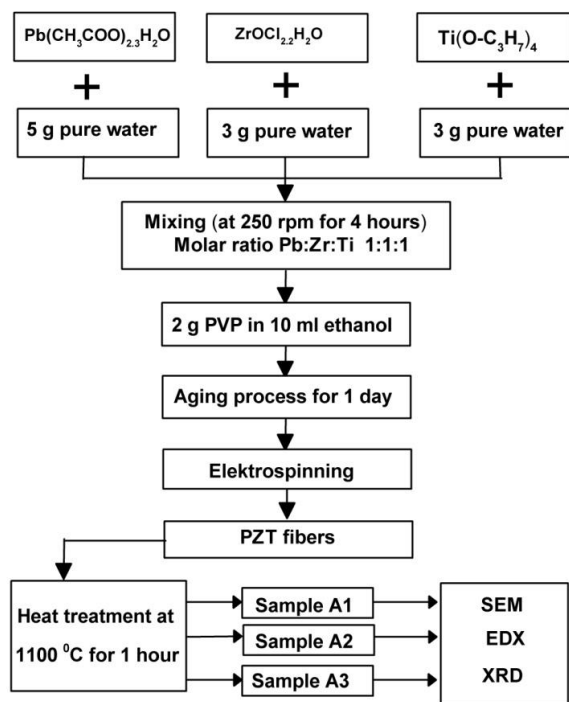


Figure 2.1 Experimental flow chart of PZT nanofibers

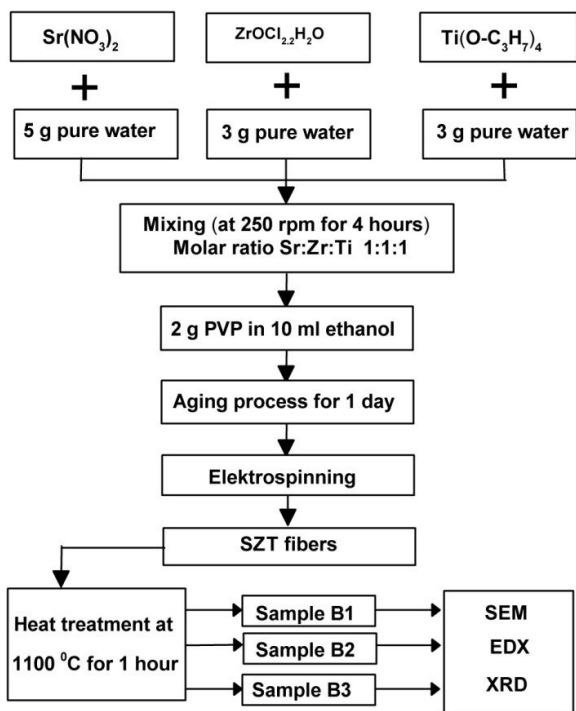


Figure 2.2 Experimental flow chart of SZT nanofibers

3. Results and Discussion

3.1. SEM Analysis of PZT Nanofibers

Figure 3.1 shows the SEM micrographs of PZT nanofibers (a)A1, (b)A2 and (c)A3. A small amount of beady structure was observed in the SEM analysis of PZT nanofibers.

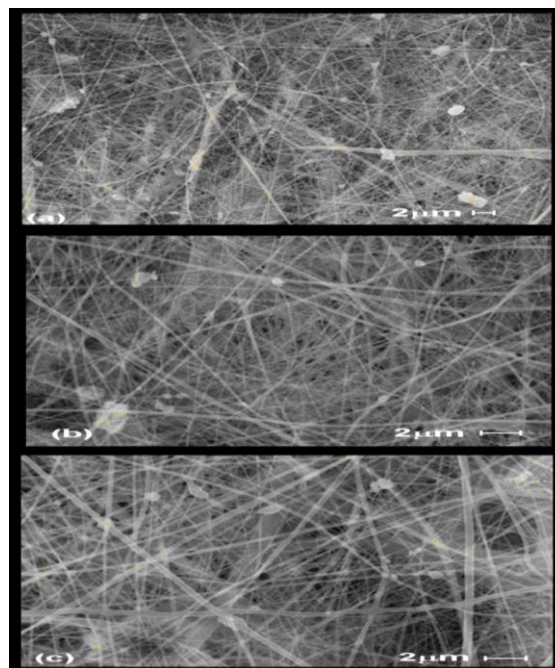


Figure 3.2. SEM Analysis of PZT Nanofibers (a)A1, (b)A2 and (c)A3.

3.2. SEM Analysis of SZT Nanofibers

SEM Analysis of SZT Nanofibers is given in Figure 3.2. A small amount of beady structure was observed in the SEM analysis of SZT nanofibers.

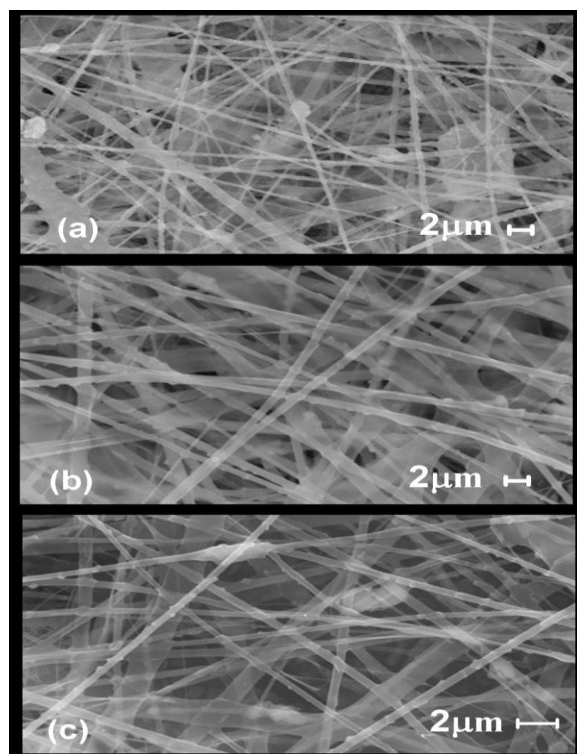


Figure 3.2. SEM Analysis of SZT Nanofibers (a)B1, (b)B2 and (c)B3.

3.3. EDX Analysis of PZT Nanofibers

Figure 3.3 shows the EDX analysis of sample A2. Results of EDX analysis of PZT nanofibers were showed that PZT

structure consisting of 9.62% titanium, 1.32% zirconium, 89.06% lead was obtained.

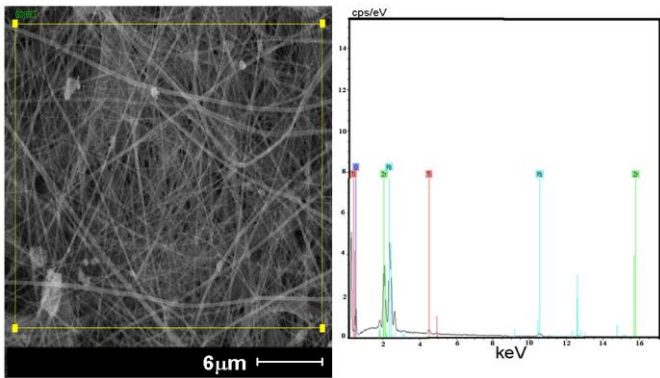


Figure 3.3. EDX Analysis of Sample A2.

3.4. EDX Analysis of SZT Nanofibers

Figure 3.4 shows the EDX analysis of sample B2. According to the EDX analysis of SZT nanofibers, it was observed that the SZT structure was also formed.

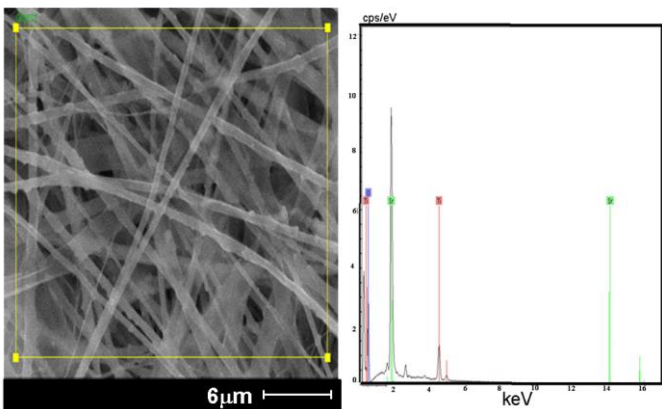


Figure 3.4. EDX Analysis of Sample B2.

3.5. Diameter Analysis of PZT Nanofibers

SEM and diameter analysis of PZT nanofibers are given in Fig. 3.5 (a)A1, (b)A2 and (c)A3 respectively. As a result of the removal of all impurities from the fiber structure after heat treatment, some decrease in the diameter of the fibers was observed.

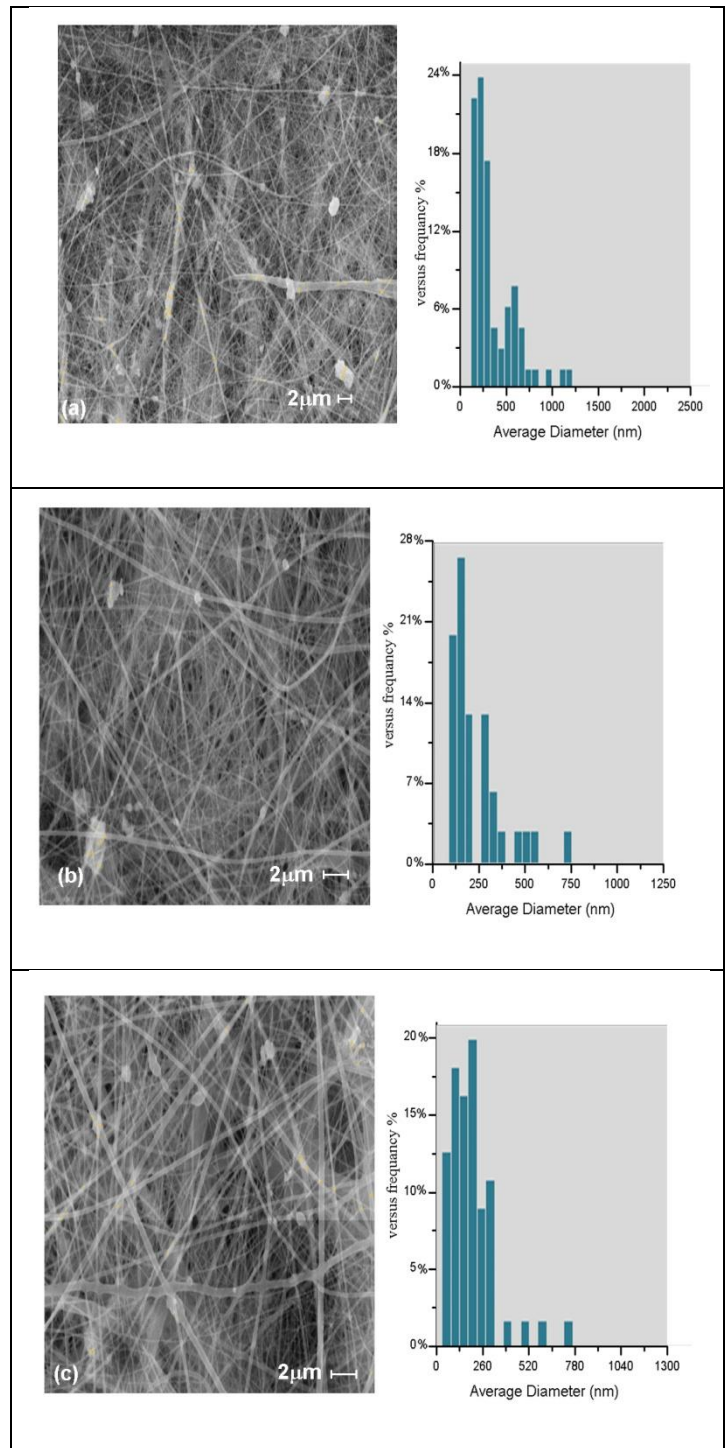


Figure 3.5. Diameter Analysis of PZT Nanofibers (a)A1, (b)A2 and (c)A3.

3.6. Diameter Analysis of SZT Nanofibers

SEM and diameter analysis of SZT nanofibers are given in Fig. 3.6 (a)B1, (b)B2 and (c)B3 respectively. As seen in Morphology and average diameters, all SZT nanofiber samples are in the form of uniform fibers, but their diameters differ. As a result of the removal of all impurities from the fiber structure after heat treatment, some decrease in the diameter of the fibers was observed.

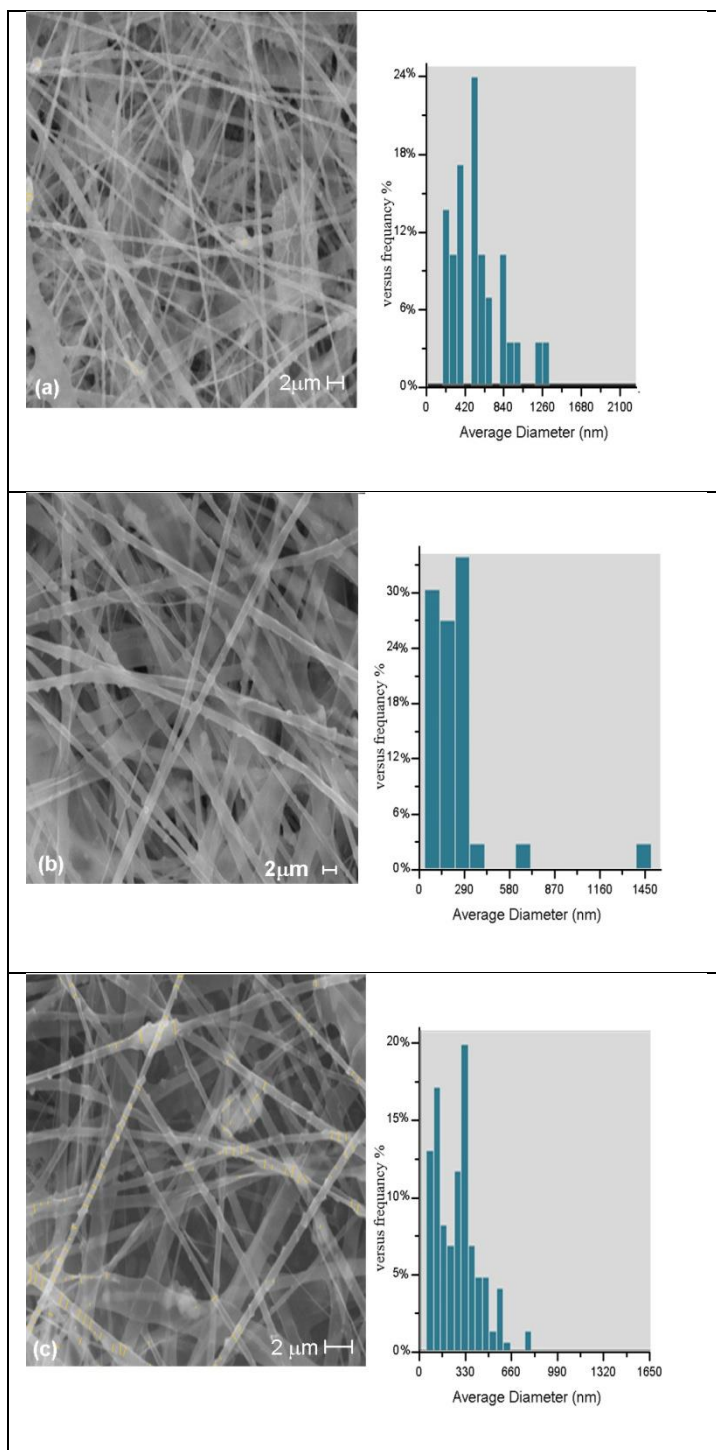


Figure 3.6. Diameter Analysis of SZT Nanofibers (a)B1, (b)B2 and (c)B3.

3.6. XRD Analysis of PZT and SZT Nanofibers

XRD patterns of the A2 and B2 electrospun fibers sintered at 1100 °C for 1 hour are seen in Fig. 3.7. As seen from Fig 3.7, XRD patterns showed that well-defined peaks, indicating crystallinity and phase formation of synthesized compounds. Perovskite structure of PZT and SZT is undisturbed during of sol-gel and electrospinning process.

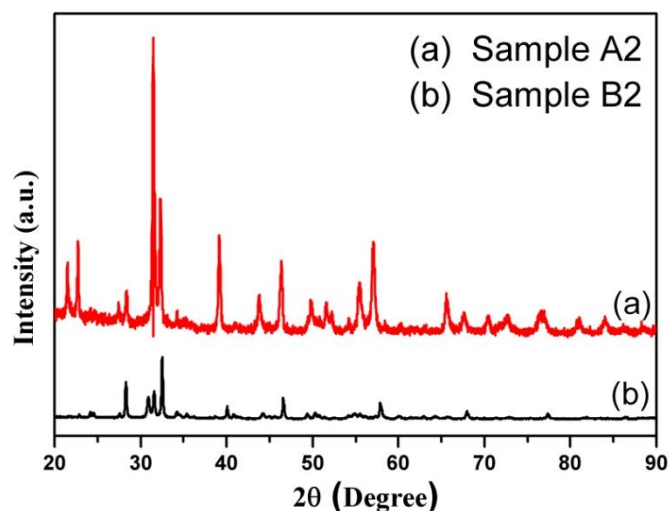


Figure 3.7. XRD patterns of nanofibers of (a) Sample A2 (b) Sample B2.

4. Conclusions and Recommendations

In this study, PZT and SZT nanofibers have been successfully obtained via by electrospinning method and positive results were obtained. The produced PZT and SZT nanofibers were calcined at 1100 °C for 1 hour. A small amount of beady structure was observed in the SEM analysis of PZT and SZT nanofibers. As a result of the removal of all impurities from the fiber structure after heat treatment, some decrease in the diameter of the fibers was observed.

Results of EDX analysis of PZT nanofibers were showed that PZT structure consisting of 9.62% titanium, 1.32% zirconium, 89.06% lead was obtained. According to the EDX analysis of SZT nanofibers, it was observed that the SZT structure was also formed. As a result of the EDX analysis, SZT structure consisting of 9.62% Titanium, 1.32% Zirconium and 89.06% Strontium was obtained. According to XRD patterns, a complete formation of PZT and SZT perovskite structure was obtained at sintering temperature of 1100 °C for 1 hour.

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