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Partially Transformation of Zinc Nitrate to Zinc Compounds on PVC Nanofibers at Low Temperature Heat Treatment and Investigation of the Products' Optical Properties

PVC Nanoliflerde Çinko Nitratın Düşük Sıcaklıkta Isıl İşlemle Kısmi Olarak Diğer Çinko Bileşiklerine Dönüşümü ve Optik Özelliklerinin İncelenmesi

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PARTIALLY TRANSFORMATION OF ZINC NITRATE TO ZINC COMPOUNDS ON PVC NANOFIBERS AT LOW TEMPERATURE HEAT TREATMENT AND INVESTIGATION OF THE PRODUCTS' OPTICAL PROPERTIES

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ABSTRACT: Zinc compounds are photoactive material and they are incorporated into the material to differentiate the optical properties of the material. PVC/$Zn(NO_3)_2$ (polyvinyl chloride /zinc nitrate) composite precursor nanofibers were produced by electrospinning by dissolving PVC and $Zn(NO_3)_2$ in the electrospinning solution together. As-spun nanofibers are treated with aqueous NaOH and then stayed at 80°C for one hour to partially convert $Zn(NO_3)_2$ to zinc compounds in/on the PVC nanofibers. Nanofibers have been investigated in terms of morphological, chemical, thermal and optical properties. An increase in transmittance values was observed at the nanofibers after thermal treatment due to the ratio of $Zn(NO_3)_2$ in the precursor PVC/ $Zn(NO_3)_2$ nanofibers, and a decrease in reflectance values was observed. The produced fine nanofibers and nanofiber coated surfaces could be potentially used to coat drapery fabrics, where controllable light transmission is required.

Keywords: Polyvinyl chloride, nanofibers, electrospinning, zinc nitrate, zinc hydroxide

PVC NANOLİFLERDE ÇİNKO NİTRATIN DÜŞÜK SICAKLIKTA ISIL İŞLEMLE KISMİ OLARAK DİĞER ÇİNKO BİLEŞİKLERİNE DÖNÜŞÜMÜ VE OPTİK ÖZELLİKLERİNİN İNCELENMESİ

ÖZET: Çinko bileşikleri fotoaktif malzemeler olup polimerler başta olmak üzere birçok malzemenin içerisine katılarak malzemelerin optik özelliklerini farklılaştırabilmektedirler. Çinko bileşiklerinin üretilen polimer nanolifin her bölgesinde bulunmasını sağlamak amacıyla çinko nitrat ($Zn(NO_3)_2$) prekürsor polivinil klorür (PVC) ile birlikte çözülerek elektro çekim metoduyla PVC/$Zn(NO_3)_2$ kompozit nanolifler üretilmiştir. Nanolifler önce sulu NaOH çözeltisiyle işlem, ardından 80^oC de fırınlanarak PVC nanoliflerdeki $Zn(NO_3)_2$ ın diğer çinko bileşiklerine dönüştürülmesi sağlanmıştır. Nanolifler morfolojik, kimyasal, termal ve optik özellikler bakımından incelenmiştir. Prekürsor PVC/$Zn(NO_3)_2$ nanoliflerdeki $Zn(NO_3)_2$ oranına bağlı olarak termal işlem sonrası nanoliflerde transmittans değerlerinde artma gözlemlenirken reflektans değerlerinde azalma gözlemlenmiştir. Üretilen ince nanolifler ve nanolif kaplı yüzeyler içerisindeki çinko bileşeni miktarına bağlı olarak ışık geçişinin kontrollü olması istenen perdelik kumaşların kaplanmasında kullanılabilirler.

Anahtar Kelimeler: Polivinilklorür, nanolif, elektroçekim, çinko nitrat, çinko hidroksit

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1. INTRODUCTION

Zinc oxide is a photoinduced active materials and has been used various application such as UV protection [1], dye synthesized solar cell [2] and photoinduced antimicrobial applications [3]. Zinc hydroxide is used for photodegredation of harmful dye molecules [4]. Zinc oxide can be produced from different zinc precursors including zinc acetate, zinc chloride and zinc nitrate [5,6]. Treatment of zinc precursor with aqueous sodium hydroxide solution with a following heat treatment leads to transformation of precursor to zinc oxide [5,6].

ZnO/PVC composite structures have been produced for various applications. Al-Taa'Y et al. investigated the optical properties of different ratio of ZnO nanoparticle loaded PVC, and reported that increasing ZnO content enhances to optical absorption and decreases the optical transmittance [7]. Enhanced transparency and antibacterial properties have been obtained from ZnO nanoparticle embedded PVC where ZnO particles covalently modified with ethylenediaminetetraacetic acid [8]. In another study, optical absorption increase and transmittance decrease with ZnO addition to PVC, and hence such materials can be used for packing due to filtration of UV and visible light [8].

Nanofibers are promising materials which have high surface area to volume ratio. High specific surface can enhance the device performance to several order of magnitude when they are used in a specific device. Nanofibers have been produced from diversity of materials such as ceramics, polymers and metals and used for very broad applications including biomedical, energy and filtration [9]. Silk fibroin [10], polyvinyle alcohol [11], and poly (acrylonitrile-co-maleic acid) [12] nanofibers have been produced via electrospinning technique.

Preparation and the usability of zinc oxide loaded polymer nanofibers have been reported by different research groups. Production of cellulose acetate-zinc oxide nanofibers via electrospinning technique and their application on optical, antibacterial and water repellency properties have been observed [13]. Growth of zinc oxide nanocrystals on electrospun cellulose acetate butyrate was examined and reported [5]. Polyvinyle chloride can ben soluble in an appropriate solvent and can be electrospun into nanofibers by using as prepared solution [14,15].

In this study, pure PVC and zinc nitrate loaded PVC nanofibers have been produced. All as-spun nanofibers were treated in aqueous NaOH with a following heat treatment process at 80°C for 1 hour to convert zinc precursor into other zinc compounds in/on PVC nanofibers. Morphological analyses were conducted by a scanning electron microscopy (SEM) and chemical analysis were carried out via a fourier transform infrared spectroscopy (FTIR). Thermal analyses were conducted with differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Optical reflectance and transmittance plots were obtained from a UV-vis-near spectrometer.

2. EXPERIMENTAL SET-UP AND PROCEDURE

2.1. Chemicals: PVC, $Zn(NO_3)_2$, dimethylformamide (DMF) and tetrahydrofuran (THF) were used and all chemicals were used as received without further purification.

2.2. Electrospinning of PVC/$Zn(NO_3)_2$ hybrid nanofibers: Schematic illustration of the preparation of PVC/$Zn(NO_3)_2$ nanofibers via electrospinning method is given in Figure 1. At first, 15 wt.% and 20 wt.% of PVC were dissolved in DMF/THF (1/1) by magnetic stirring in an ambient condition for 24 hours. Then, 5, 15 and 30 wt.% of $Zn(NO_3)_2$/PVC solutions were prepared by adding $Zn(NO_3)_2$ in the previously prepared PVC solution. As-prepared solution was filled in a plastic syringe and stayed on a micropump system for a lineer solution feding. A high voltage was applied to the metal needled of the syringe to eject solution droplet to the grounded collector system. As a consequence of the ejection of the solution droplet from the needle to collector, PVC and PVC/$Zn(NO_3)_2$ nanofibers were formed on the grounded metal surface (Figure 1). Solvent in the solution was evaporated during the formation of nanofibers and dry nanofibers were collected on the surface.

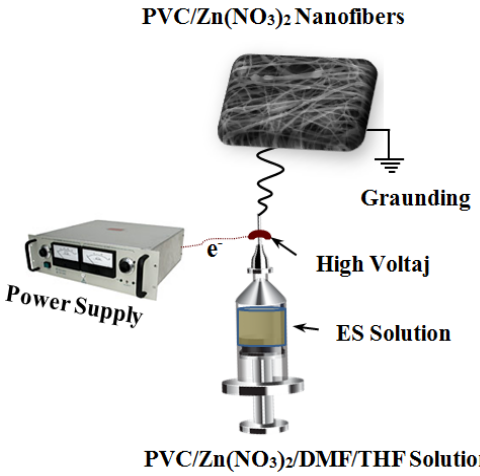

Figure 1. Schematic illustration of the preparation of PVC/$Zn(NO_3)_2$ nanofibers via electrospinning method.

2.3. Synthesis of Zinc compounds in PVC nanofibers: As-spun PVC and PVC/$Zn(NO_3)_2$ nanofibers were stayed in aqueous NaOH (0.8 wt %.) solution for 12 hours with a following rinsing with water to remove excess NaOH. Then, nanofibers were put in a oven and stayed at 80 °C for 1 hour to convert $Zn(NO_3)_2$ to other zinc compounds in PVC nanofibers.

3. CHARACTERIZATIONS

The morphology of PVC and PVC/$Zn(NO_3)_2$ nanofibers were analyzed using a ZEISS EVO 40 scanning electron microscopy (SEM). In order to reduce charging during SEM analysis, samples were first set on a SEM sample holder and then they coated with gold-palladium (about 100 Å of thickness) using a BAL-TEC SCD005 sputter coater. Then the samples were put in the SEM chamber for analysis. 20 kV acceleration voltage was used for SEM analysis. Attenuated total reflection Fourier transform infrared (ATR-FTIR) spectra of PVC and PVC/$Zn(NO_3)_2$ nanofibers were collected for chemical analysis in the wavenumber range of 4000 to 400 cm^{-1} at room temperature. ATR-FTIR spectra were obtained on a Bruker TENSOR37 instrument. DSC measurement was Elmer Pyris 1, TGA measurement was Perkin Elmer DSC Pressure, and optical

analyses were conducted with UV-3600 Plus Spektrofotometer. X-ray diffraction characterization was conducted by Rigaku Ultima IV X-Ray diffractometer.

4. RESULTS AND DISCUSSIONS

High resolution SEM images of the as-spun PVC and $Zn(NO_3)_2$ /PVC nanofibers were conducted as in Figure 2. As-spun nanofibers were uniform and form 3D network structures on the collected form.

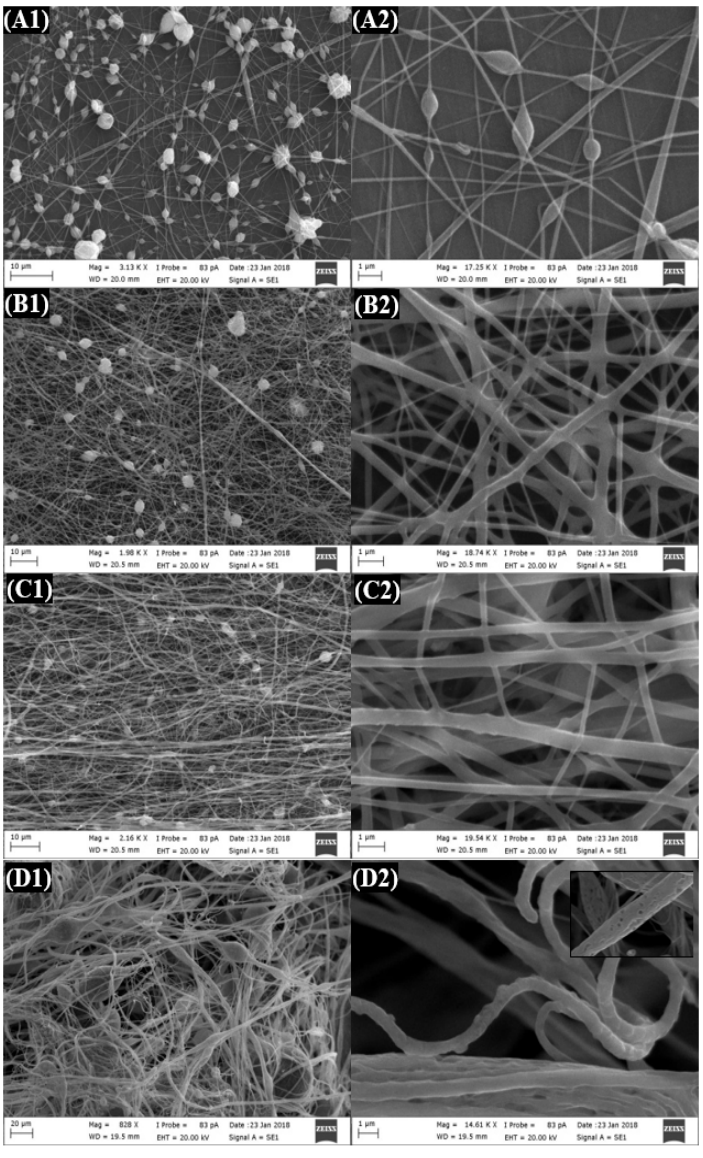

Figure 2. SEM images of PVC and PVC/$Zn(NO_3)_2$ nanofibers: (A1, A2): pure PVC, (B1, B2): 15 wt. % $Zn(NO_3)_2$/PVC, (C1, C2): 30 wt. % $Zn(NO_3)_2$/PVC, and (D1, D2): 50 wt. % $Zn(NO_3)_2$/PVC.

SEM images of the pure PVC NFs produced from 15 wt. % polymer solution in DMF/THF (1/1) were given in Figure 2 (A1, A2). PVC/$Zn(NO_3)_2$ nanofibers where $Zn(NO_3)_2$ to PVC ratio were 15, 30 and 50 wt. % were given in Figure 2 (B1, B2), Figure 2 (C1, C2) and Figure 2 (D1, D2). Bead-on-a-string morphology was seen for as-spun pure PVC nanofibers (Figure 2 (A1, A2)). When zinc nitrate was added to the PVC nanofibers this defect structure is reduced and more uniform nanofibers were obtained. PVC nanofibers were collected regularly on the metal collector, but addition of $Zn(NO_3)_2$ made PVC/ $Zn(NO_3)_2$ nanofibers difficult to collect on the collector surface. Nanofibers spread out with increasing $Zn(NO_3)_2$ content in PVC/$Zn(NO_3)_2$ nanofibers and hence nanofibers were collected near the collector with a more oriented form. As seen from Figure 2, nanofiber diameter increases with increasing zinc nitrate concentration. Up to %30 PVC/ $Zn(NO_3)_2$ in the prepared solution, totally transparent solutions were obtained but %50 sample was blurry. Encapsulated $Zn(NO_3)_2$ were seen in %30 PVC/$Zn(NO_3)_2$ sample (Figure 2C) and some porous structures with encapsulated structure were observed at 50wt. % $Zn(NO_3)_2$/PVC nanofibers (Figure 2D). It was so difficult to obtain $Zn(NO_3)_2$/PVC nanofibers when content $Z_n(NO_3)_2$ was 50 wt. %, so working sample ratios were selected as pure PVC, 5, 15 and 30 wt. % of PVC/ $Zn(NO_3)_2$ for the following studies.

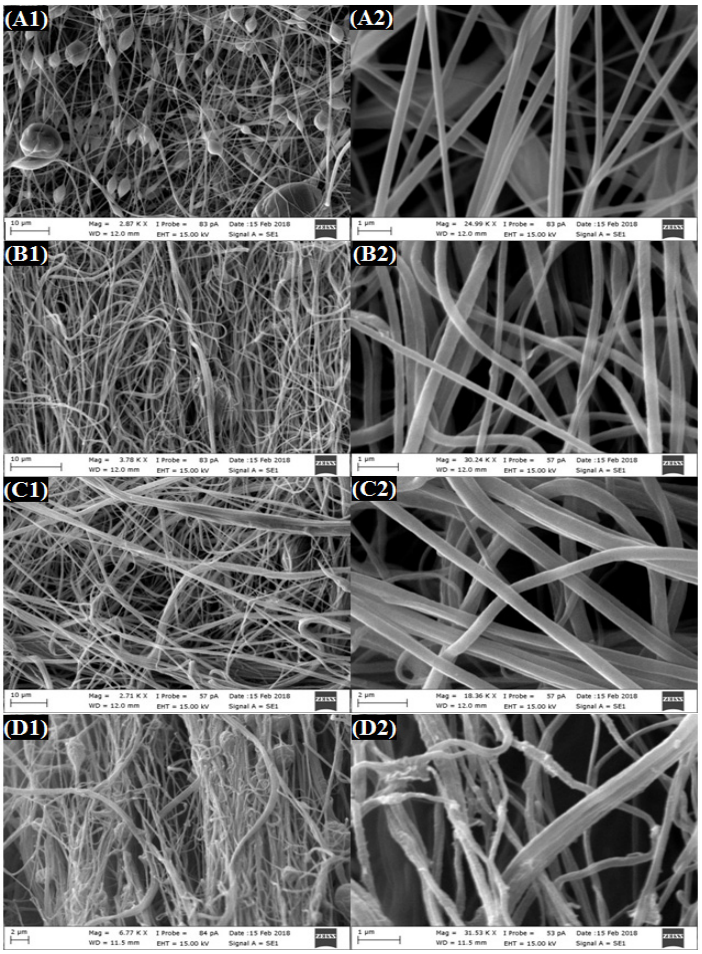

Figure 3. SEM images of aqueous NaOH treated PVC and PVC/Zn $(NO_3)_2$ nanofibers: (A1, A2): pure PVC, (B1, B2): 5 wt. % $Zn(NO_3)_2$/PVC, (C1, C2): 15 wt. % $Zn(NO_3)_2$/PVC, and (D1, D2): 30 wt. % $Zn(NO_3)_2$/PVC.

SEM images of PVC and PVC/$Zn(NO_3)_2$ nanofibers that stayed in aqueous 0.8 wt %. of NaOH solution for 12 hours and rinsed with deionized water several times is demonstrated in Figure 3. Comparing with as-spun nanofibers as in Figure 2., no obvious difference has been detected. Some nanoparticulation is seen in 30 wt. % sample (Figure 3. D1, D2).

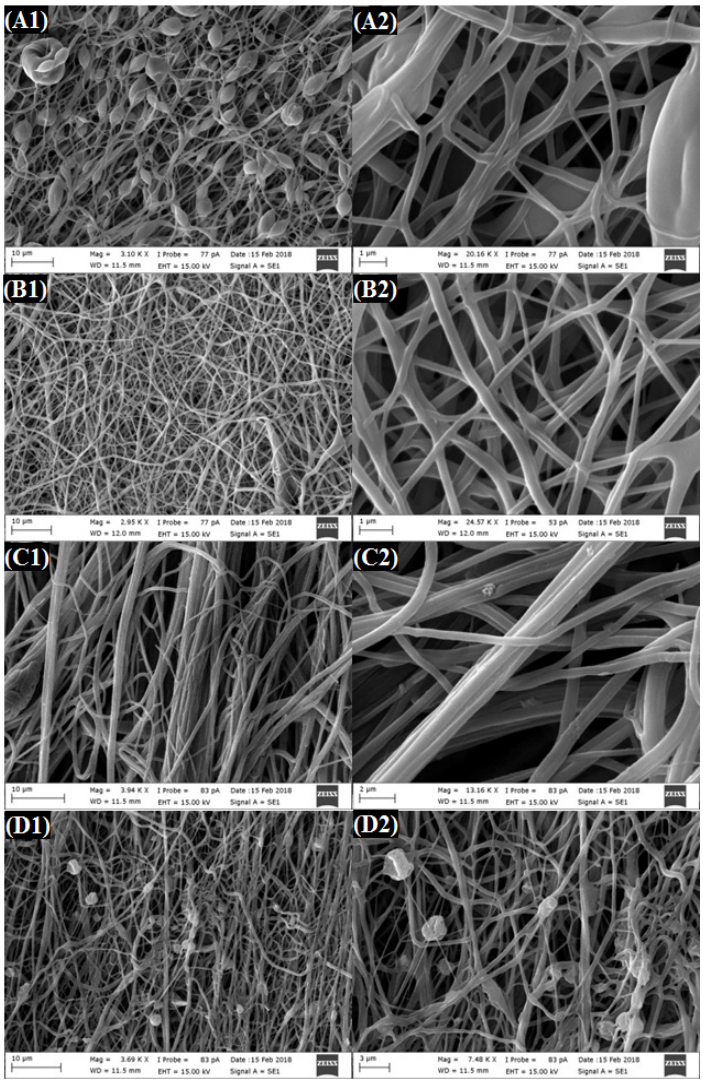

Figure 4. SEM images of heat treated (at 80 ^{o}C for 1 hour) of aqueous NaOH treated PVC and PVC/$Zn(NO_3)_2$ nanofibers : (A1, A2): pure PVC, (B1, B2): 5 wt. % $Zn(NO_3)_2$/PVC, (C1, C2): 15 wt. % $Zn(NO_3)_2$/PVC, and (D1, D2): 30 wt. % $Zn(NO_3)_2$/PVC.

SEM images of heat treated PVC and PVC/$Zn(NO_3)_2$ nanofibers which treated with aqueous 0.8 wt. % of NaOH solution for 12 hours and then treated at 80 ^{o}C for 1 hour is given in Figure 4. No obvious difference has been detected at pure PVC nanofibers by comparing as-spun and NaOH treated samples (In Figure 2. A1, A2, and Figure 3. A1, A2). On the other hand, particulate form of transformed zinc compounds are clearly seen on PVC nanofibers at 15 and 30 wt. % samples (Figure 4. C2, D1 and D2). Nanofiber uniformity has not been destroyed by depending on aqueous NaOH and following heat treatment processes.

Figure 5A. shows ATR-FTIR spectra of pure PVC and PVC/ $Zn(NO_3)_2$ composite nanofibers with different $Zn(NO_3)_2$ ratio, respectively. In pure PVC nanofibers (Figure 3A), the characteristic abosorption peaks of PVC were detected for C-Cl stretching mode observed at 842 cm^{-1}, trans CH wagging mode at 968 cm^{-1} and cis CH wagging mode at 615 cm^{-1}[16,17]. CH stretching at 2914 cm^{-1}, CH-rocking at 1251 cm^{-1} and CH_2 deformation at 1330 cm^{-1} were observed [16,17]. The presence of NO_3^{-1} increases the peak intensities between 1220-1370 cm^{-1} [18]. After NaOH and heat treatment these peak intensities decreased (Figure 5B, 5C) since some NO_3 has been removed as a result of NaOH treatment and a following rinsing. Characteristic bands between 400-500 cm^{-1} correspond to Zn-O stretching (Figure 5C), and the intensities increased by increasing $Zn(NO_3)_2$ ratio in PVC/ $Zn(NO_3)_2$ nanofibers [19,20].

DSC measurement results of pure PVC and PVC/ $Zn(NO_3)_2$ composite nanofibers were demonstrated in Figure 6A. Glass transition temperature (Tg) of PVC was detected at 83,73 ^{o}C [21,22]. Since addition of $Zn(NO_3)_2$ reduces the PVC molecular mobility, Tg was increased a little bit at 5 wt. % sample [21]. But, with increasing $Zn(NO_3)_2$ content, Tg was reduced since it may be related to structural damage and moving away of PVC molecules from each other that cause easy mobility of the molecular chains [22].

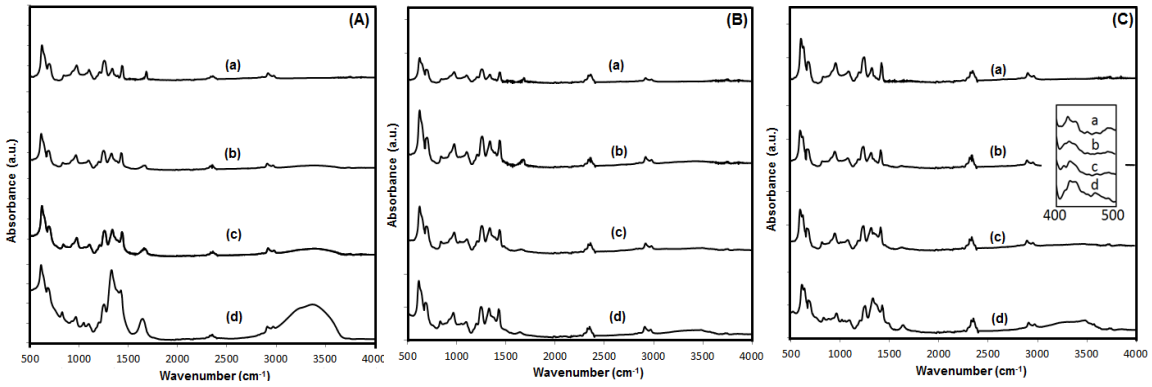

Figure 5. ATR-FTIR spectra of nanofibers: (A) as-spun, (B) aqueous NaOH treated as-spun, and (C) at 80^{o}C 1 hour heat treated nanofibers in (B). (a) pure PVC, (b) 5 wt. % $Zn(NO_3)_2$/PVC, (c) 15 wt. % $Zn(NO_3)_2$/PVC, and (d) 30 wt. % $Zn(NO_3)_2$/PVC.

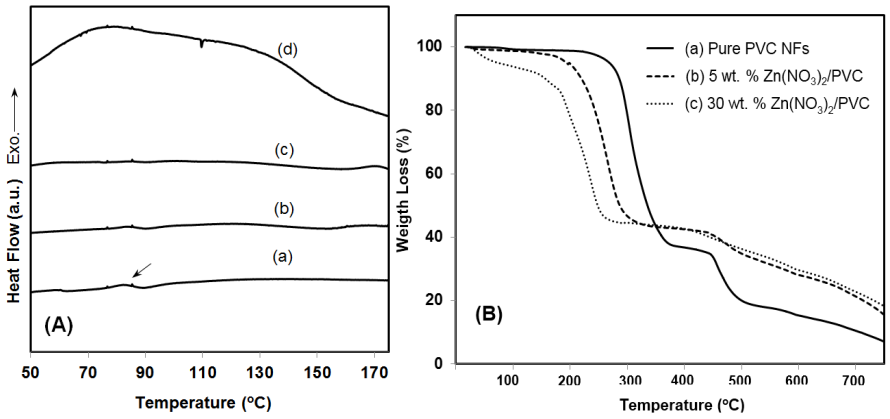

Figure 6. (A) DSC: (a) pure PVC, (b) 5 wt. % Zn(NO3)2/PVC, (c) 15 wt. % Zn(NO3)2/PVC, and (d) 30 wt. % $Zn(NO_3)_2$/PVC; and (B) TGA : (a) pure PVC, (b) 15 wt. % $Zn(NO_3)_2$/PVC, and (c) 30 wt. % $Zn(NO_3)_2$/PVC nanofibers.

In order to observe the thermal properties of nanofibers TGA measurements also were conducted and the results are demonstrated in Figure 6B. Weight loss between 0-100 ^oC can be related to removal of water and other vapour came from electrospinning solution that holded by nanofibers. Two main mass losses around 251-372 ve 451-501 ^oC were seen. First one can be related to removel of Cl as HCl from pure PVC and PVC/ $Zn(NO_3)_2$ nanofibers [15,21]. After removel of Cl from PVC, polyene was obtained [21]. Polyene production was occurred at lowere temperature with addition of zinc nitrate to PVC nanofibers. Second main weith loss is smaller tha first one and corresponds to removal of gas that produced from polyene degredation [21]. Residual material after TGA measurement increases with increasing zinc precursor. Addition of zinc nitrate decreased the degradation temperature of nanofibers.

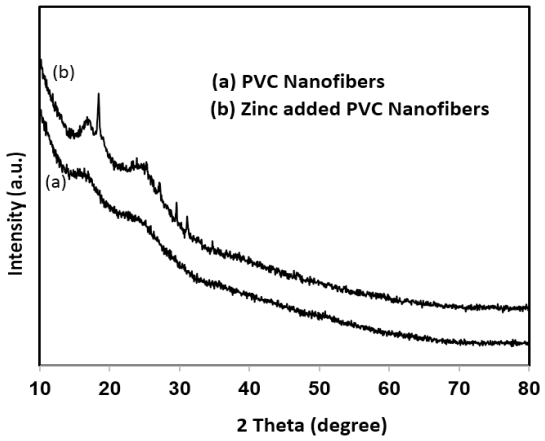

Figure 7. X-Ray diffraction (XRD) patterns of (a) PVC and (b) NaOH and heat treated PVC/$Zn(NO_3)_2$ nanofibers.

Transformation of the zinc nitrate to any crystalline zinc compound was observed with X-ray diffraction measurement (Figure 7.). Broad XRD peaks for PVC were detected around 16.79 and 23.57^o can be attributed to partially crystalline of PVC nanofibers [23]. The slight increase in the intensities of these peaks can attributed to addition of zinc compound and heat treatment enhanced the crystallinity of PVC nanofiber. Since corresponding ZnO peaks are not presented at the XRD patterns [18,19], it is very difficult to mention that zinc nitrate was transformed to well crystallize zinc oxide structure in this study. Generally transformations of zinc nitrate to zinc oxide occur at higher temperatures than the temperature carried out in this study. On the other hand some strong XRD peaks were detected around 15.74, 18.42, 25.88, 27.09,29.55, 31.08, and 34.07^o which probably come from transformed zinc nitrate hydroxide ($Zn(OH)(NO_3)(H_2O)$) and zinc hydroxide ($Zn(OH)_2$) [24]. It was considered that the hydrophobic character of PVC does not allows the aqueous NaOH solution readily penetrate into PVC/$Zn(NO_3)_2$ nanofibers. Accordingly, some minor zinc hydroxide formation might have occurred.

In the literature, it was reported that when ZnO was added to PVC, UV absorption increased and transmittance was decreased [7,8]. Optical reflectance and transmittance plots of nanofibers produced in this study were demonstrated in Figure 8. In general, when zinc nitrate was added in PVC nanofibers reflectance values was decreased depending on $Zn(NO_3)_2$ content (Figure 7B). This trend continued after NaOH and heat treatment of nanofibers (Figure 8A). On the other hand, transmittance values of nanofibers was decreased with increasing $Zn(NO_3)_2$ content in PVC (Figure 8C). Optical properties of nanofibers coated cotton fabrics also investigated after aqueous NaOH and thermal treatment at 80 ^oC for 1 hour. As seen from Figure 8D, low content of $Zn(NO_3)_2$ was not effect remarkably, but 30 wt. % sample coated cotton fabric show enhanced transmittance values (Figure 8D). Li et al. reported that nanofiber orientation enhances the optical transmittance [25]. As seen from SEM images in this study, when $Zn(NO_3)_2$ content increase in PVC nanofibers, more uniform nanofibers were obtained. The increase of transmittance can be related to increase of fiber orientation. Also, porous mat structure can be the other reason of the light transmittance. In another study, Bolarinwa et al. reported that polyvinyl acetate and zinc acetate dihydrate nanofibers when precursor nanofibers were heat treated enhanced optical transmittance [26]. The low thickness of nanofiber mat can be the other reason of the enhanced transmittance values. On the other hand, low temperature (80^oC) heat treatment could not be enough to convert zinc nitrate to an appropriate zinc oxide for better light absorption by the composite nanofibers and also confirmed by XRD data in Figure 7. On contrast, reflectance values were decreased by increasing zinc precursor contents PVC nanofibers.

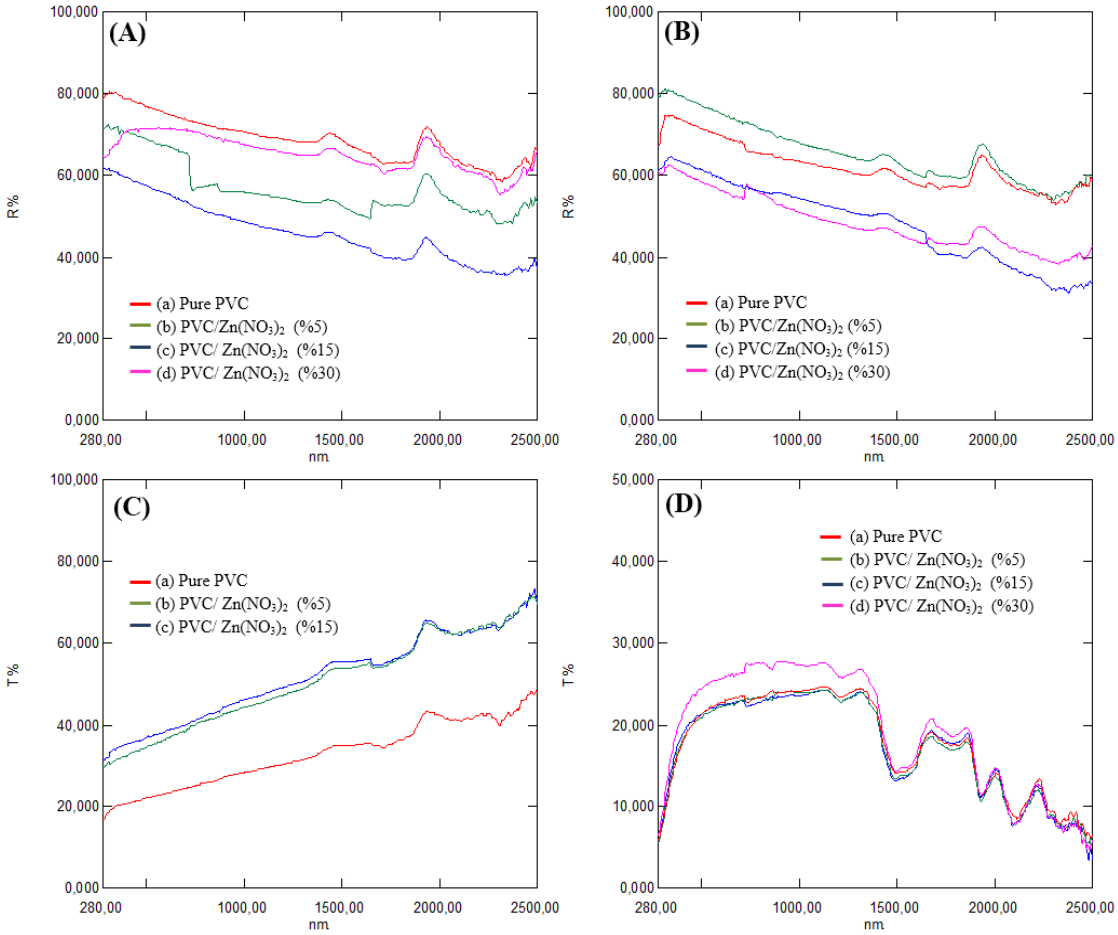

Figure 8. (A) Reflectance plots of NaOH and heat treated PVC/ $Zn(NO_3)_2$ nanofibers: a) pure PVC, b) 5 wt. % $Zn(NO_3)_2$/PVC, c) 15 wt. % $Zn(NO_3)_2$/PVC, and d) 30 wt. % $Zn(NO_3)_2$/PVC; (B) Reflectance plots of PVC/$Zn(NO_3)_2$ nanofibers: a) pure PVC, b) 5 wt. % $Zn(NO_3)_2$/PVC, c) 15 wt. % $Zn(NO_3)_2$/PVC, and d) 30 wt. % $Zn(NO_3)_2$/PVC; (C) transmittance plots of NaOH and heat treated PVC/ $Zn(NO_3)_2$ nanofibers: a) pure PVC, b) 5 wt. % $Zn(NO_3)_2$/PVC and c) 15 wt. % $Zn(NO_3)_2$/PVC; transmittance plots of NaOH and heat treated PVC/ $Zn(NO_3)_2$ nanofibers coated cotton fabrics: a) pure PVC, b) 5 wt. % $Zn(NO_3)_2$/PVC, c) 15 wt. % $Zn(NO_3)_2$/PVC and d) 30 wt. % $Zn(NO_3)_2$/PVC

5. CONCLUSIONS

PVC/$Zn(NO_3)_2$ composite nanofibers were produced via electrospinning procedure by using different PVC to $Z_n(NO_3)_2$ ratio. Bead-on-a-spring nanofibers morphologies observed at neat PVC nanofibers with the used solution concentration. When $Zn(NO_3)_2$ exist in the nanofibers defect nanofiber structure transforms to more uniform fiber morphology and nanofibers diameters increase. Zinc precursor was converted to other zinc compounds after a proper aqueous NaOH and a following heat treatment process. Zinc nitrate in PVC nanofibers transformed to zinc nitrate hydroxide and zinc hydroxide after conducting aqueous NaOH and heat treatment processes. Nanofiber uniformity has not been lost due to aqueous NaOH and following heat treatment processes. TGA measurements revealed that addition of zinc nitrate into PVC nanofibers decreased the degradation temperature of the nanofibers. Residual material after TGA measurement increases with increasing zinc precursor content in the nanofibers. Optical transmittance increased and reflectance decreased depending on the ratio of precursor $Zn(NO_3)_2$ in PVC/$Zn(NO_3)_2$ nanofibers.

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