TEKSTİL VE MÜHENDİS

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The Synergic Effects of Electrospinning Solution Properties on Formation of Polyvinyl Chloride (PVC) Fibers

Elektroeğirme Çözelti Özelliklerinin Polivinil Klorür (PVC) Liflerinin Oluşumu Üzerindeki Sinerjik Etkileri

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Online Erişime Açıldığı Tarih (Available online):30 Haziran 2022 (30 June 2022)

Bu makaleye atıf yapmak için (To cite this article):

Mukaddes Şevval ÇETİN, Aybüke Sultan DEMİREL, Ozan TOPRAKÇI, Hatice Aylin KARAHAN TOPRAKÇI (2022): The Synergic Effects of Electrospinning Solution Properties on Formation of Polyvinyl Chloride (PVC) Fibers**,** Tekstil ve Mühendis, 29: 126, 42-49.

For online version of the article: https://doi.org/10.7216/1300759920222912601

TMMOB Tekstil Mühendisleri Odası
UCTEA Chamber of Textile Engineers
Tekstil ve Mühendis
Journal of Textiles and Engineer

Yıl (Year) : 2022/2
Cilt (Vol) : 29
Sayı (No) : 126

Araştırma Makalesi / Research Article

THE SYNERGIC EFFECTS OF ELECTROSPINNING SOLUTION PROPERTIES ON FORMATION OF POLYVINYL CHLORIDE (PVC) FIBERS

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Gönderilme Tarihi / Received: 19.01.2022
Kabul Tarihi / Accepted: 20.05.2022

ABSTRACT: In this study, the optimization process for polyvinyl chloride fibers (PVC) fabrication by electrospinning was performed. The effects of solvent system, and polymer concentration were investigated in terms of fiber formation by considering solution viscosity and conductivity. In order to do this, three different solvent systems were used including dimethylformamide (DMF), tetrahydrofuran (THF), and DMF/THF (50/50) mixture. Polymer concentrations were prepared at various concentrations as 8, 10, 15, and 20 wt%. The viscosity and conductivity of the solutions were measured as a function of the polymer concentration. The conditions were kept constant as 0.2 mL h^{-1}, 15 cm, and 15kV for feed rate, needle to collector distance, and applied voltage, respectively. Solvent type, solution conductivity, polymer concentration, and solution viscosity were determined as the critical factors for electrospinnability of PVC solution. The most homogeneous fiber formation was obtained PVC/DMF/THF system and DMF was found effective for spinnability in terms of its positive contribution on solution conductivity.

Keywords: Polyvinyl chloride (PVC), electrospinning, nanofibers

ELEKTROEĞİRME ÇÖZELTİ ÖZELLİKLERİNİN POLİVİNİL KLORÜR (PVC) LİFLERİNİN OLUŞUMU ÜZERİNDEKİ SİNERJİK ETKİLERİ

ÖZ: Bu çalışmada, elektroeğirme yöntemi ile polivinil klorür (PVC) liflerinin üretilme prosesi optimize edilmiştir. Çözgen sistemi ve polimer konsantrasyonu lif oluşumuna etki eden değişkenler olarak incelenmiştir. Bunun için dimetil formamid (DMF), tetrahidrofuran (THF) ve DMF/THF (50/50) karışımı olmak üzere üç farklı solvent sistemi kullanılmıştır. Polimer konsantrasyonları ağırlıkça %8, 10, 15 ve 20 olarak belirlenmiştir. Çözeltilerin viskozitesi ve iletkenliği polimer konsantrasyonunun fonksiyonu olarak ölçülmüştür. Besleme hızı, iğne-kolektör mesafesi ve uygulanan voltaj için koşullar sırasıyla 0.2 mL h^{-1}, 15 cm ve 15kV olarak sabitlenmiştir. Çözücü tipi, çözelti iletkenliği, polimer konsantrasyonu ve çözelti viskozitesi PVC çözeltisinin eğrilebilirliği açısından kritik faktörler olarak bulunmuştur. En homojen lif oluşumu PVC/DMF/THF sisteminden elde edilmiştir ve DMF, çözelti iletkenliği ve eğrilebilirliğe olan olumlu katkısı açısından etkili bulunmuştur.

Anahtar kelimeler: Polivinil klorür (PVC), elektroeğirme, nanolifler

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DOI: *https://doi.org/10.7216/1300759920222912601* *www.tekstilvemuhendis.org.tr*

1. INTRODUCTION

Polyvinyl chloride (PVC) is one of the common polymers because of its properties such as high mechanical strength, high elastic modulus, abrasion resistance, and toughness. In addition to these, PVC can be plasticized and it provides tunable mechanical properties [1]. It can be used for various industries including coating [2–4], electronics [5], construction [6–8], pipe [9], automobile [10,11], textiles [12], shoe [13], biomedical [14], and food [15,16]. In these applications PVC powder can be converted into sheets, profiles, coating layers, injection molded parts, films, membranes, fibers, nonwovens by melt or solution-based processes. However, its low thermal stability leads to usage of thermal stabilizer during the melt process. Because of thermal sensitivity solution based processes are preferred for membrane, coating, and fiber production [1].

Electrospinning is a common method for the fabrication of ultrafine or nanofibers from polymer solution. Although various products can be obtained at the end of electrospinning, common geometry is the porous and pliable nonwoven. Since this process has many variables, the fiber and mat morphology is tunable [17–20]. In the literature, the relationship between PVC fiber morphology and applied voltage, polymer concentration, solvent type, needle-collector distance were investigated and given in a review study [21]. One of the first studies of electrospun PVC fibers was carried out by Lee et al. [22]. In their work, dimethylformamide (DMF) and tetrahydrofuran (THF) and DMF/THF mixtures were used and effects of polymer concentration, DMF/THF ratio, applied voltage, and needle-collector distance were investigated. Although this work was very comprehensive, viscosity-solution conductivity relation was not investigated [22]. Similar study was also carried out by Phatcharasit et al. [23]. In another study, Carrizales et al. fabricated the PVC fibers and characterized thermal and mechanical properties [24]. In that study, DMF/THF mixture was used and process conditions were kept constant [24]. Medeiros et al. investigated the relative humidity factor on PVC fiber morphology [25]. In that study, DMF was used as the solvent and relationship between fiber morphology and humidity ratio were investigated under constant conditions. As reported in the study, at higher relative humidity values, the average fiber diameter increased and surface porosity changed [25]. Lackowski fabricated electrospun PVC nonwoven filter and investigated their performance [26]. In that study, DMF/THF mixture was used as the solvent system and all processing conditions were kept constant and only one fibrous nonwoven mat was fabricated. They showed the filtering performance of PVC nonwoven [26]. Tarus et al., prepared PVC fibers and mechanical properties of the PVC fibers were investigated [27]. Zulfi et al., recycled PVC from the pipes and used DMF, THF, and dimethylacetamide (DMA). 4 different polymer concentrations were determined for all solvents and fiber formation was investigated [28]. Rivero et al., used THF/DMF as the solvent system. Two different polymer solutions were electrospun with two different feed rate, under two different applied voltage and multifunctional protective PVC fibers were fabricated with tunable properties [29]. Le et al. used flat plate and rotary drum collectors for the electrospinning of PVC fibers. Six different drum rotation speeds were determined. Rotational speed was found to effect the fiber diameter and orientation [30]. Uslu et al. fabricated PVC nanofibers [31]. THF/DMF was used as the solvent and one type of nanoweb was fabricated, that web was used as a reinforcement for epoxy composites [31]. In another study Hong et al., fabricated electropun PVC mats from DMF based solutions. In the first step PVC fiber formation was investigated based on polymer concentration. In the second step they have blended PVC with soy protein and investigated fiber formation by considering solution viscosity and conductivity [32].

As given in the above review, in order to optimize fiber formation of PVC, mostly polymer concentration [32], process conditions [21,22,29], collector type [30], environmental conditions [25], solvent type [24] were investigated and spinnability of PVC/THF/DMF was found better compared to other solvent systems [21,22]. However, solution viscosity and solution conductivity were not considered, and these properties were not compared for PVC/THF and PVC/DMF systems previously. For that reason, in this work, 3 different solvent systems as DMF/THF, DMF and THF were used, spinnability and fiber formation were investigated based on polymer concentration, solvent type, solution conductivity and solution viscosity for the first time in the literature. This study is of significance for fabrication of PVC fibers with tunable morphologies.

2. MATERIALS AND METHODS

2.1. Materials

Polyvinyl chloride (PVC), K value of 58, was in the form of white powder that produced by suspension polymerization. Dimethylformamide (DMF) and tetrahydrofuran (THF) were bought from Merck. The conductivity of THF was very low and DMF was used to increase the electrical conductivity of the electrospinning solution. All materials were used as received.

2.2. Preparation of the spinning solutions

In order to investigate the solvent type and polymer concentration, electrospinning performance and fiber morphology, solutions were prepared at different polymer concentrations (8, 10, 15, and 20 wt %) with various solvent systems as given in Fig. 1 and Table 1.

For each solution, the solvents were DMF, THF, and DMF/THF with the ratio of 50/50. As given in Fig. 1, twelve solutions were prepared by using a magnetic stirrer at 40 °C and 800 rpm until clear solutions were obtained (around 24 h).

2.3. Electrospinning

The fabrication of nanofibers via electrospinning process was performed by using a pump system (New Era Syringe Pump System, NE-300) and a high voltage power supply (Zhengzhou TAINUO Film Materials Co, P303-2ACDF0), as illustrated in Fig. 2. During the electrospinning experiments, the temperature and relative humidity were set to 25 ± 2 °C and %60 RH. In order to observe the effect of solvent type on electrospinning performance and fiber morphology, the feed rate, needle to collector distance, and applied voltage were kept constant. These values were determined after the optimization study under various conditions. For all spinning solutions, feed rate, needle to collector distance, and applied voltage were 0.2 mL h^{-1}, 15 cm, and 15 kV respectively.

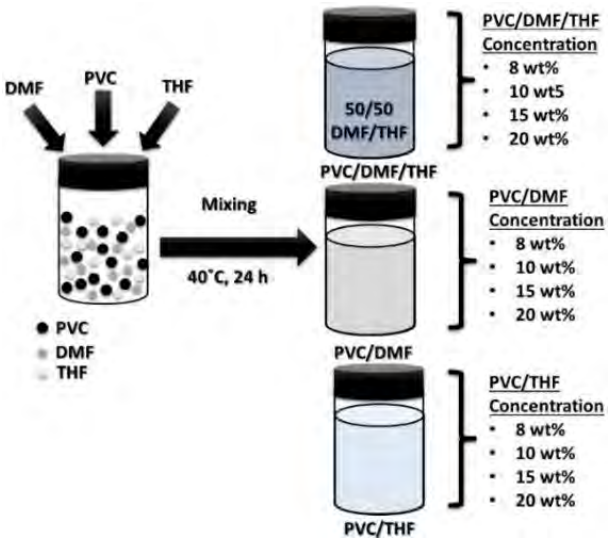

Figure 1. Preparation and classification of the electrospinning solutions

Table 1. Sample code and content of the electrospinning solutions

Sample Code	Solvent	Polymer Concentration (wt %)
S1	DMF/THF	8
S2	DMF/THF	10
S3	DMF/THF	15
S4	DMF/THF	20
S5	DMF	8
S6	DMF	10
S7	DMF	15
S8	DMF	20
S9	THF	8
S10	THF	10
S11	THF	15
S12	THF	20

2.3. Characterization

The viscosity of electrospinning solutions was measured by using a viscometer, Brookfield DV2T. The measurements were carried out at 30 rpm with a # 3 spindle under 25°C, %60 RH. The measurements were repeated three times for all samples and the average values were calculated.

The electrical conductivity of the electrospinning solutions was measured by using pH/conductivity meter, VWR PC 5000 L. For all solutions, measurements were carried out three times under 25 °C, %60 RH and average values were calculated.

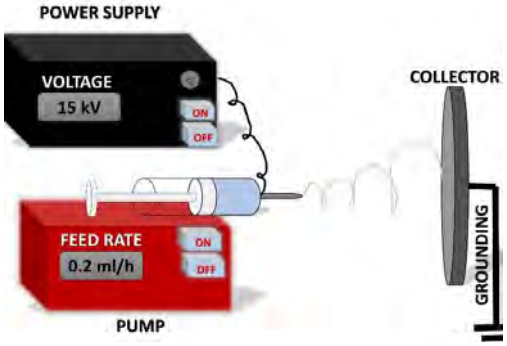

Figure 2. Electrospinning set-up

The optical images of the electrospun fibers were taken by a light microscope (AmScope 40X-2500X LED Digital Binocular Compound Microscope). Fiber diameter distribution of the samples were determined by using Image J software. Before the analysis, samples were removed from aluminum foil.

Scanning electron microscopy (SEM) analysis was performed under 20 kV at Yalova University Central Research Laboratory (FEI Inc., Inspect S50 SEM). Prior to analysis samples were sputter coated by Au/Pd alloy. Coating thickness was around 3-6 nm. The SEM images can be seen as the insets of fiber diameter distribution histograms.

3. RESULTS AND DISCUSSION

Electrospinning performance and fiber morphology is governed by many factors and viscosity is accepted as one of the most significant parameters because of its direct effect on spinnability. The viscosity of the spinning solutions can be seen from Table 2.

Table 2. Viscosity (V), normalized viscosity (NV) and conductivity (C) and fiber formation status of PVC spinning solutions

Code	V (cP)	NV (cP/cP)	C (mS cm-1)	Fiber Formation
S1	36.67	1	1.7	No
S2	73.33	2	2.4	Fiber+Bead
S3	280	7.6	1.6	Yes
S4	2410	65.7	1.9	Yes
S5	110	1	9.8	No
S6	216.7	2	9.9	No
S7	733	6.6	10.0	No
S8	13160	120	9.4	Clogging
S9	30	1	0	No
S10	56.67	1.9	0	No
S11	263.3	8.8	0	Fiber+Bead
S12	2050	68.3	0.1	Yes

In Fig. 3, viscosity and normalized viscosity values were given as a function of polymer concentration in the linear scale and $\log_{10}$ scale. Normalized viscosity (NV) values were calculated for each group based on the solvent system. In order to evaluate the relative change in viscosity, viscosity values were divided by viscosity of 8 wt% PVC containing spinning solution ($\eta/\eta_{8\ wt\%}$). Viscosity of the solution is governed by polymer concentration and solvent type. Concentration effects the viscosity basically by polymer-polymer interaction and entanglement ratio. In our case, as expected at higher concentrations polymer-polymer interaction increased because of higher entanglement ratio led to higher viscosity for all solvent systems as reported previously [32].

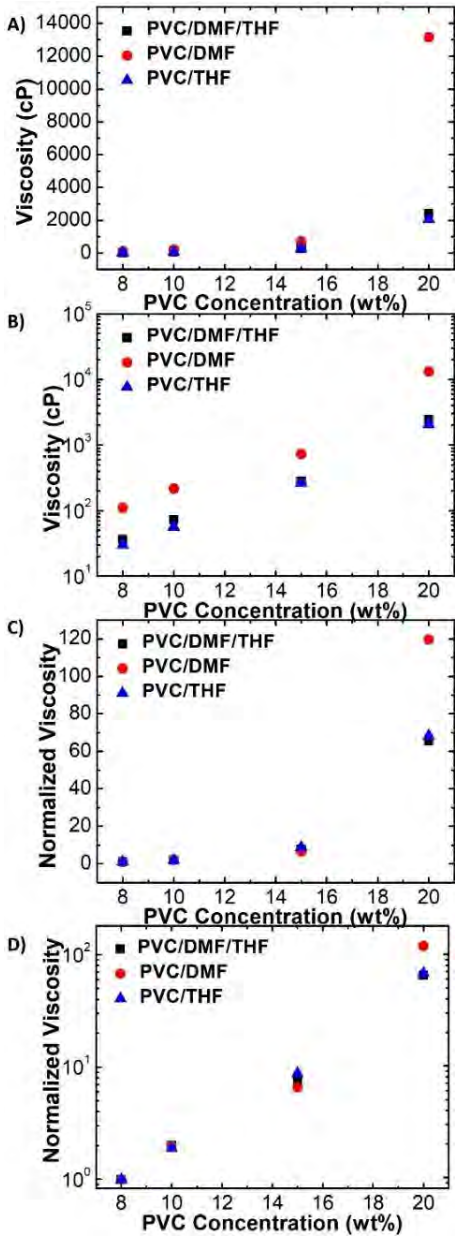

Figure 3. a) Viscosity values of the spinning solutions b) viscosity values of the spinning solutions represented in log scale, c) normalized viscosity values, and d) normalized viscosity values of the spinning solutions represented in log scale

When the solvent systems were considered, the viscosity values for same polymer concentrations can be given as PVC/DMF > PVC/DMF/THF > PVC/THF. While DMF based solutions showed the highest viscosity, THF based solutions showed the lowest. Although both solvents are polar, and can dissolve PVC, THF was reported to show better dissolution performance compared to DMF. As given by Grause et al., under same conditions while THF dissolved PVC completely (good solvent), DMF led to high level of swelling (rather poor) [33]. Since the level of interaction between PVC and solvent directly affects the swelling ratio of the polymer chains and viscosity of the solution, that behavior could be related to polymer-solvent interactions. Tager et al., reported that solvents that have higher solubility performance (good solvents) can lead to breaking up the bundle structure and that will result in lower viscosity at concentrated solutions [34,35].

As given in Table 1 and Fig. 4, solvent system was found significant in terms of conductivity of the spinning solution. The conductivity of DMF/THF, DMF, THF were measured as 0.6, 7.9, 0 mS cm^{-1} respectively. Parallel with that, the conductivity values of solutions for same polymer concentrations were determined as PVC/DMF > PVC/DMF/THF > PVC/THF. Incorporation of DMF into THF led to increase in conductivity regardless of the polymer concentration. As previously reported by many authors [21,22], DMF has a higher conductivity value compared to THF and DMF/polymer systems can be accepted as polyelectrolyte. The difference between two solvents is basically caused by structure, dielectric constant, and dipole moment of the solvents. The level of interaction between THF molecules were reported to be higher compared to DMF molecules. That directly affects the charge distribution in the solution and polymer-solvent interaction. In the case of lower interaction between molecules, it might be advantageous during electrospinning by providing higher charging density. Although both solvents are polar, dielectric constants of DMF and THF were given as 36.7 and 7.6 (at 25 °C), respectively. The dipole moment value of DMF (3.8 D) was reported to be higher compared to THF (1.7 D) [36]. However, there was no direct relationship between conductivity and polymer concentration according to our outcomes. For THF based solutions, conductivity showed almost no change. On the other hand, for DMF and DMF/THF based solutions conductivity increased up to 10 wt% concentration, then decreased. At higher polymer concentrations, higher amount of solvent was assumed to be interacted with the polymer and the conductivity of the system decreased. The increase in conductivity up to 10 wt% PVC concentration might be caused by various factors. As known, solution conductivity is governed by many factors including presence of the ionic species in the system, solvent type, mixing ratio of the solvents, additives, impurities, polymer type, molecular weight of polymer, polymer concentration, and purity. As previously mentioned, samples were used without any purification and obtained results might be caused by above

mentioned reasons. In order to determine the exact reason for change in conductivity, components of the system should be well-known. While salts, surfactants and fillers can be evaluated as additives; impurities are undesired species those might be caused by any process related with the fabrication of the polymer, solvent or additives (polymerization, synthesis, catalysts, separation, transportation etc.) [37,38].

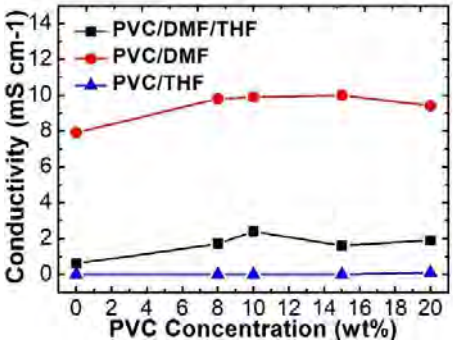

Figure 4. Conductivity values of spinning solutions

In order to observe the effects of solvent type and polymer concentration, samples were analyzed by an optical microscope and SEM. The average fiber diameter and bead size values were determined by using Image J software and corresponding histograms were drawn. Digital, optical microscope and SEM images, fiber diameter and bead size distribution histograms and average fiber diameter, bead size of S1-S4 can be seen from Fig. 5 and 6, respectively.

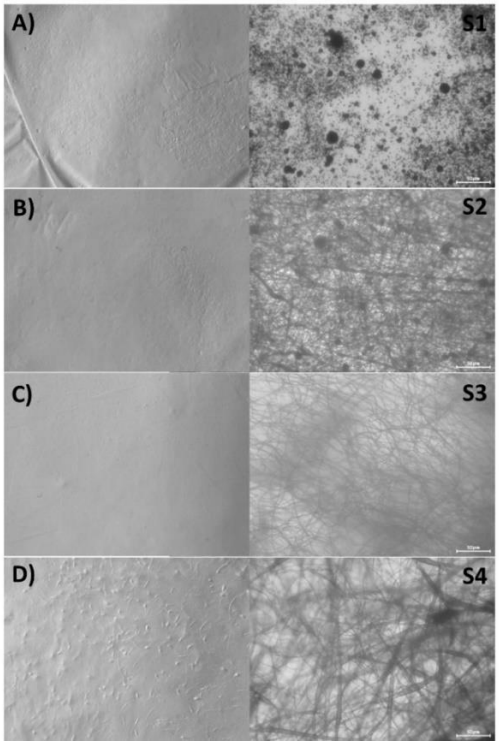

Figure 5. Digital and optical microscope images of a) S1, b) S2, c) S3, and d) S4

As can be seen from Fig. 5a, fiber formation was obtained for 10, 15 and 20 wt% PVC concentration for DMF/THF solvent system. By increasing the polymer concentration the viscosity regime probably shifted from semi-dilute unentangled regime to semi-dilute entangled regime [32]. At 8 wt% the viscosity of the solution was not high enough to resist applied electrical force and solution sprayed onto the collector without any fiber formation. The average bead size was determined as 5.10 µm. At 10 wt% both fiber and bead morphology were observed (Fig. 5b). The average bead size and fiber diameter values were 8.51 µm and 1.89 µm, respectively. Fiber morphology was observed for both 15 and 20 wt% PVC containing solutions. As obvious from the diameter distribution histograms (Fig. 6), although S4 had slightly higher conductivity, S3 had more homogeneous fiber distribution than S4. As known higher solution conductivity generally leads to better spinning performance [21,33]. In our case viscosity of the solution probably showed more dominant effect than conductivity. Solution viscosity of S4 was relatively high (8 times) than S3.

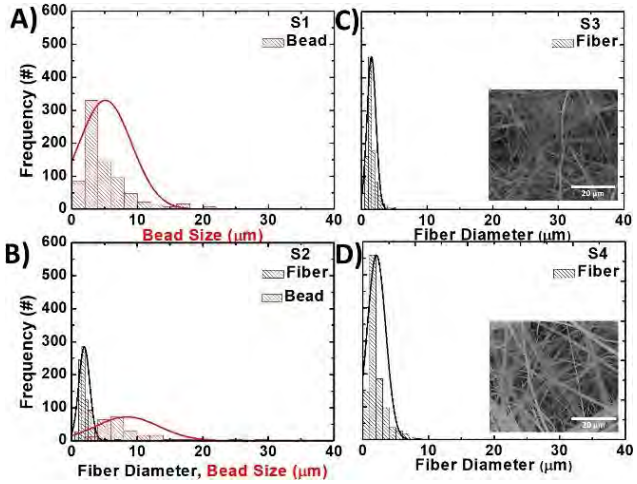

Figure 6. Fiber diameter and/or bead size distributions of a) S1, b) S2, c) S3, and d) S4

Increase in viscosity led to increase in fiber diameter. Also, that much increase probably caused instability during electrospining and led to coarser fiber distribution with less uniformity (Fig. 6c and d) [32].

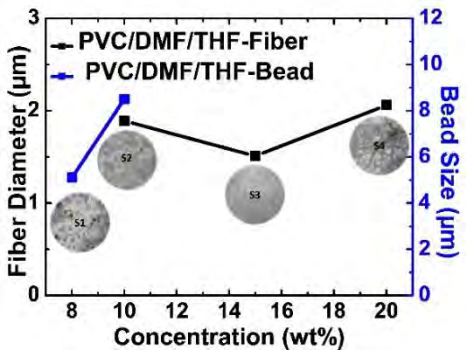

Figure 7. Average fiber diameter and/or bead size of S1, S2, S3, and S4

Digital, optical microscope and SEM images, fiber diameter and bead size distribution histograms and average fiber diameter, bead size of S5-S7 can be seen from Fig. 8-10, respectively. S8 solution could not be electrospun. As obvious from Fig. 8, continuous fiber formation was not obtained for PVC/DMF solution regardless of the polymer concentration [25,32]. For 8 wt% PVC/DMF solution beads were observed but diameter distribution could not be determined by our optical microscope because of its extra small size.

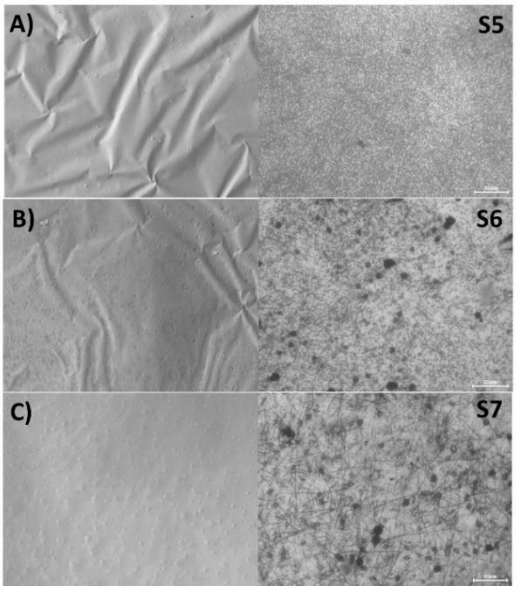

Figure 8. Digital and optical microscope images of a) S5, b) S6, c) S7

S5 and S6 samples were full of beads and bead size of S5 was relatively lower than S6. Although viscosity seemed to be good for spinning bead/fiber mixture was obtained for 15 wt% PVC/DMF.

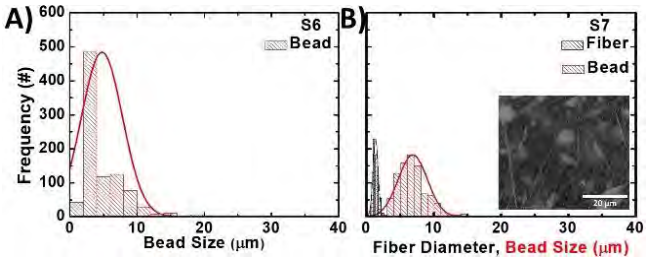

Figure 9. Fiber diameter and/or bead size distributions of a) S6 and b) S7

The spinning was not carried out at 20 wt% because of high viscosity. As mentioned previously, PVC-DMF interaction led to higher solution viscosity, clogging of the needle and spinning did not occur since applied voltage was not high enough for the jet formation and spinning.

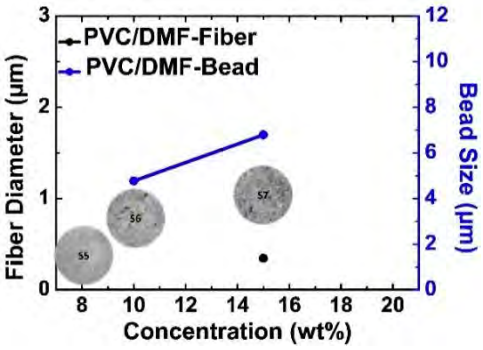

Figure 10. Average fiber diameter and/or bead size of S6 and S7

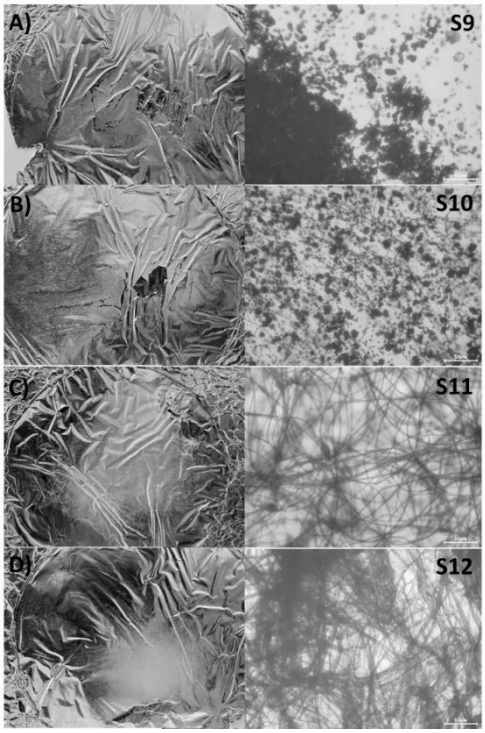

Figure 11. Optical microscope images of a) S9, b) S10, c) S11, and d) S12

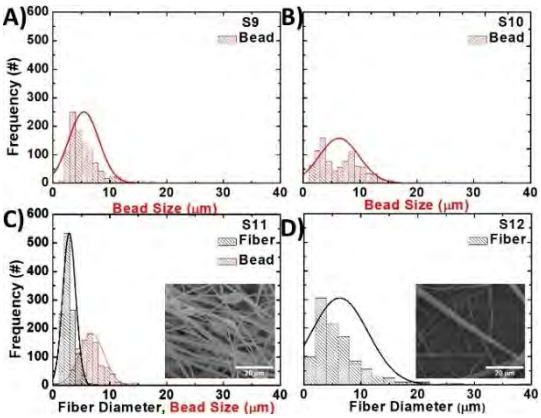

Figure 12. Fiber diameter and/or bead size distributions of a) S9, b) S10, c) S11, and d) S12

Digital, optical microscope and SEM images, fiber diameter and bead size distribution histograms and average fiber diameter, bead size of S9-S12 can be seen from Fig. 11-13, respectively. As obvious from Fig. 11 and 12, only bead formation was obtained at 8 and 10 wt% PVC concentration probably because of low solution viscosity and conductivity.

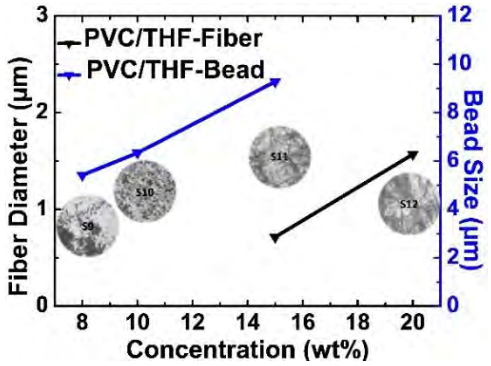

Figure 13. Average fiber diameter and bead size of a) S9, b) S10, c) S11, and d) S12

For 15 wt% PVC/THF solution both beads and fibers were observed but needle clogging problem prevent the continuous fiber production. Fiber formation was obtained for 20 wt% PVC/THF solution, however the needle clogged continuously, and it was hard to collect fibers. That was probably caused by lower boiling of THF. While THF has boiling point around 65 ºC, DMF has around 150 ºC [39].

4. CONCLUSIONS

Electrospun PVC fibers were fabricated by using various spinning solutions. Solvent type and polymer concentration were determined as the variables for this study. DMF, THF, and DMF/THF (50/50) mixtures were used and 8, 10, 15 and 20 wt% PVC solutions were prepared for each system. Electrospinning was performed under 15/15 kV/cm with 0.2 ml h^{-1} feeding rate. From the outcomes of this study given above, the following conclusions can be drawn:

Electrospinning of PVC/DMF: PVC/DMF solutions showed the highest conductivity and highest viscosity values compared to other solutions. That was caused by high conductivity of DMF and PVC-DMF interaction. However, high conductivity was not found enough for fiber formation. At 8, 10 wt% PVC concentrations, only bead formation was observed. In this case, viscosity of the solution was not sufficient for a continuous jet formation. At 15 wt% both beads and fibers formed. Although solution viscosity was high enough, conductivity-viscosity balance could not be obtained. At 20 wt% the viscosity was too high and applied voltage was not high enough for the jet formation and spinning. This also led to clogging of the needle.

Electrospinning of PVC/THF: PVC/THF solutions showed the lowest conductivity and highest viscosity values compared to other solutions. That was caused by low conductivity of THF and PVC-THF interaction. At 8, 10 wt% PVC concentrations, only bead formation was observed. In this case, viscosity and conductivity of the solution were not sufficient for a continuous jet and fiber formation. At 15 wt% both beads and fibers formed. At 20 wt% fiber formation was observed because of high viscosity. However, needle clogged almost every 20-30 s, applied voltage was not sufficient for the continuous jet formation and spinning.

Electrospinning of PVC/DMF/THF: PVC/DMF/ THF solutions showed the conductivity and viscosity values between PVC/DMF and PVC/THF solutions. Incorporation of DMF led to increase in solution viscosity and conductivity. Like DMF and THF based systems, at 8 wt% PVC concentration, bead formation was observed. That was probably caused by low solution viscosity and conductivity. At 10 wt% PVC concentration unlike DMF and THF based solutions, fibers were fabricated with beads. At 15 wt% perfect PVC fibers were spun without beads. That was determined as the optimum conductivity, polymer concentration and viscosity for the electrospinning of PVC under given conditions. At 20 wt% PVC concentration unlike DMF and THF based solutions, fibers were fabricated. However, homogeneity and uniformity of the fibers were not as good as 15 wt% PVC concentration. Fiber diameter distribution was wider with higher standard deviation.

As summarized above, DMF and THF were not found effective under given conditions for a homogeneous PVC electrospinning process and fiber formation. Incorporation of DMF into THF led to increase in conductivity, charge storage capacity, and viscosity of the solution and led to formation of homogeneous PVC fibers. In addition to these, the disadvantages caused by lower boiling point of THF can be compensated by DMF addition that slowed down the solvent evaporation. Future work will focus on fabrication of PVC nanocomposite fibers based on outcomes of the present study.

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