Avrupa Bilim ve Teknoloji Dergisi
Özel Sayı, S. 121-128, Kasım 2020

Araştırma Makalesi

www.ejosat.com ISSN:2148-2683

European Journal of Science and Technology
Special Issue, pp. 121-128, November 2020

Research Article

Effect of Processing Conditions on the Electrospinning Behavior of Polyvinylpyrrolidone with Lower Toxicity Solvents

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(International Symposium on Multidisciplinary Studies and Innovative Technologies (ISMSIT) 2020 – 22-24 October 2020)

(**DOI:** 10.31590/ejosat.819593)

ATIF/REFERENCE: Şenol, N. & Akkoyun, Ş. (2020). Effect of Processing Conditions on the Electrospinning Behavior of Polyvinylpyrrolidone with Lower Toxicity Solvents. *European Journal of Science and Technology*, (Special Issue), 121-128.

Abstract

Due to its properties such as biocompatibility, water solubility and stability, polyvinylpyrrolidone (PVP) becomes more and more attractive for biomedical applications. This polymeric material is used in various applications such as pharmaceutical aid, complexing agent or solubilizer. PVP nanofibers are often produced using PVP solutions in solvents with high toxicity such as dimethylformamide (DMF). The aim of this study is to investigate the effect of process parameters on the electrospinning behavior of polyvinylpyrrolidone in solvents with lower toxicity such as dimethylsulfoxide (DMSO) or ethanol. Therefore, solutions of PVP in ethanol, PVP in DMSO or PVP in binary solvent systems such as DMSO/ethanol or DMSO/acetone were prepared and electrospun. The effect of process parameters such as voltage, flow rate, tip-to-collector distance were examined. A solution parameter, the polymer concentration was also considered. The morphology and diameter of the electrospun nanofibers were characterized by scanning electron microscopy (SEM). The effect of the solution viscosity was also questioned. Nanofibers with a homogeneous cylindrical morphology were obtained in the case of PVP in ethanol solutions for a polymer concentration of 7 wt.%. The process parameters were: a voltage of 15kV, tip-to-collector distance of 15 cm and a flow rate of 1.25 mL/h. PVP in DMSO solutions didn't allow the obtention of solid nanofibers on the collector where a wetness zone appears. This shows that the solvent could not evaporate quickly. A wetness was also observed with PVP solutions prepared using binary solvent systems where a more volatile solvent such as ethanol or acetone was used.

Keywords: electrospinning, polyvinylpyrrolidone, DMSO, nanofiber, binary solvents.

Proses şartlarının polivinilpirolidon polimerinin düşük toksisiteli solventler ile elektroeğirilme davranışı üzerindeki etkisi

Öz

Biyouyumluluk, suda çözünürlük ve kararlılık gibi özellikleri sayesinde polivinilpirolidon (PVP) biyomedikal uygulama alanlarında günden güne artan bir ilgi görmektedir. Polimerik bir malzeme olan PVP farklı uygulamalarda farmasötik yardımcı madde, kompleks oluşturucu ya da çözücü olarak kullanılmaktadır. PVP nanolifleri genellikle dimetilformamid (DMF) gibi toksisitesi yüksek çözücüler ile hazırlanmış PVP çözeltilerinden üretilmektedir. Bu çalışmanın amacı toksisitesi düşük seviyede olan çözücüler kullanılarak hazırlanan PVP çözeltilerinden nanolif elde etmek ve üretim süreci şartlarının polivinilpirolidon polimerinin düşük toksisiteli çözücülerde hazırlanmış çözeltilerinin elektroeğirilme davranışı üzerindeki etkisini araştırmaktır. Bu anlamda etil alkol, dimetilsülfoksit (DMSO) ve DMSO/etil alkol ve DMSO/aseton sistemlerinden oluşan ikili çözücüler ile karıştırılmış PVP çözeltileri hazırlanmış ve elektroeğirilmiştir. Çalışma kapsamında voltaj, akış hızı, iğne ucu-toplayıcı mesafesi gibi üretim süreci parametrelerinin etkisi araştırılmıştır. Ayrıca, çözeltinin özelliklerinin etkisini incelemek adına, polimerin derişiminin etkisi de incelenmiştir. Elektroeğirilmiş nanoliflerin morfolojileri ve çapları taramalı elektron mikroskobu (SEM) ile incelenmiştir. Çözeltinin viskozitesinin etkisi de ayrıca incelenmiştir. Etil alkol ile hazırlanmış % 7 oranında PVP içeren çözeltilerin elektroeğirilmesi sonucunda homojen silindirik nanolifler elde edilmiştir. Kullanılan üretim süreci parametreleri şu şekilde verilebilir: nanoliflerin üretiminde 17.5 kV'luk bir voltaj uygulanmıştır, iğne ucu-toplayıcı mesafesi 15 cm olarak sabitlenmiştir ve 1.25 mL/s oranında bir akış hızı kullanılmıştır. DMSO ile hazırlanan PVP

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çözeltileri ile katı nanolifler elde edilememiş ve toplayıcı üzerinde bir ıslaklık gözlemlenmiştir. Bu durum aslında DMSO'nun yeterince hızlı bir şekilde uçamadığını göstermektedir. Bu sebeple daha uçucu çözücüler olan etil alkol ve aseton ile ikili çözücü sistemler ile PVP çözeltileri hazırlanıp elektroeğirilmiştir. İkili çözücü sistemleri ile de DMSO çözeltileri ile olduğu gibi toplayıcı üzerinde bir ıslaklık gözlemlenmiştir.

Anahtar Kelimeler: elektroeğirme, polivinilpirolidon, DMSO, nanolif, ikili çözücü.

1. Introduction

Due to their small pore size and high aspect ratio or surface area to volume ratio, nanofibers present superior material performances. Different production techniques have been developed to use the advantages of nanofibers in various application areas. Several methods exist for the production of polymer nanofibers such as drawing, template synthesis, phase separation, self-assembly and electrospinning. With the increasing popularity of nanotechnology in recent years, the use of polymers as ultrafine fibers remains important (Huang, Zhang, Kotakic, & Ramakrishna, 2003). Electrospinning, allows the production of nanofibers from polymer solution or melt, and complex and continuous three-dimensional fiber can be obtained. This technique is at the forefront due to its simple and effective application compared to other fiber production methods. High production rate and low cost are among its advantages. This technique consist in the production of continuous fibers using polymer melt or polymer solution. The device consists of a feeding pump with a metal tip syringe, a high voltage supply and a grounded conductive collector where the fibers are collected. The collector can be used vertically or horizontally (Rogina, 2014).

In electrospinning a high voltage is applied on the metallic needle of the syringe containing the polymer solution during the pumping through the metallic collector. The electric field induces the formation of a conical shape (Taylor cone) at the tip of the needle. When the electrostatic forces overcome the polymer surface tension forces, the charged jet is ejected from the Taylor cone, and uniaxial deformation occurs towards the grounded collector. Thus, with the whipping of the formed jet, the solvent evaporates, and solid continuous fibers are formed on the collector (Rogina, 2014), (Unnithan, R.S., & Kim, 2015), (Reneker & Chun, 1996).

It is important to examine the process parameters that affect fiber morphology and diameter (Frenot & Chronakis, 2003). Homogeneous fibers with desired diameter and morphology can be obtained by changing these parameters (Unnithan, R.S., & Kim, 2015) (Alghoraibi & Alomari, 2018) (Haider, Haider, & Kang, 2018).

The molecular weight of the polymer has important effects in forming fibers from the viscoelastic jet. If a lower concentration of polymer is used, beaded structures are formed instead of continuous fibers. The increase of the molecular weight makes the fiber morphology homogeneous and more stable. However, very high molecular weights cause the formation of micro and flat fibers instead of nanofibers. Moreover, if the molecular weight increeases, the viscosity increases and the evaporation rate of the solvent can be reduced, and thus, the dry fibers can be collected on the collector (Unnithan, R.S., & Kim, 2015), (Koski, Yim, & Shivkumar, 2004).

The choice of the solvent is one of the most important parameters depending on the application area targeted and influencing fiber diameter and fiber morphology. The solvent must completely dissolve the polymer. Solvents with high evaporation rates are desired as they facilitate the dry fiber formation. Weakly volatile solvents cause the fibers to remain wet and retard their drying, involving the formation of beaded nanofibers. In some cases, a second solvent is added to the polymer solution in order to provide fast evaporation of solvent with different evaporation rates (Haider, Haider, & Kang, 2018).

In recent years, polyvinylpyrrolidone (PVP) found application in different areas. Its properties such as non-toxicity, good physiological biocompatibility, stable chemical structure, good solubility in water and various organic solvents, and good physical properties make PVP more and more attractive. PVP has applications in various fileds such as biomedical applications, pharmaceutical industry, optical and electrical equipments, membranes, adhesives, cosmetics and many environmental and engineering applications. For biomedical applications, studies are focused on the use of PVP nanofibers in wound and burn dressing, fast-dissolving drug membranes, core shell nanofibers for wound healing and the use of magnetic core shell fibers for cellular imaging (Teodorescu & Bercea, 2015).

Therefore, the aim of this study is to investigate the effect of process parameters on electrospinning behavior of PVP in solvents with lower toxicity such as dimethylsulfoxide (DMSO) or ethanol. The effective electrospinning of nanofibers from PVP in DMSO solution would be important especially for biomedical applications. Although very few studies on needleless electrospinning of PVP are present in the literature (Wortmann, et al., 2019), to our knowledge, no study exist on the electrospinning of this polymer/solvent system with metallic needle. Thus, experiments were realized with ethanol and DMSO as solvents. In order to improve the volatility of DMSO, binary solvent systems composed of DMSO/ethanol or DMSO/acetone were also investigated.

2. Materials and Methods

2.1. Materials

The polymer used in this study is polyvinyllpyrrolidone, PVP (K85-95, Mw ~1 300 kg/mol, Acros Organics). Ethanol with a density of 0.789 g/cm^3 and molar mass of 46,07 g/mol was used as solvent (96% purity, Dr. Derman). The other solvents used are dimethyl sulfoxide (DMSO) with a density of 1.1 g/cm^3 and a purity >99% (Merck) and acetone (BIRPA Kimya, Purity: 99.5%, Density: 0.791-0.793 g/cm^3).

2.2. Preparation of polymer solutions

PVP in ethanol of different concentrations (between 5 wt.% and 14 wt.%) were prepared in order to determine the optimum polymer concentration. For this purpose, PVP powder was dissolved into ethanol under magnetic stirring for 3h at ambient temperature. PVP in DMSO, PVP in DMSO/Acetone solvent mixture at a 9:1 ratio and PVP in DMSO/Ethanol solvent mixture at a ratio of 8:2 were also prepared using the same method detailed above.

2.3. Fabrication of PVP nanofibers by electrospinning

An in-house electrospinning device was used for the fabrication of nanofibers. A schematic representation of the electrospinning process is given in Figure 1. The process starts by introducing the polymer solution into the syringe. Then, the solution is pumped through the grounded metallic collector. When a high voltage is applied on the metallic needle, a polymer solution whipping jet evolves through the collector. During their trajectory, the nanofibers solidify as the solvent evaporates and solid nanofibes are gathered on the collector.

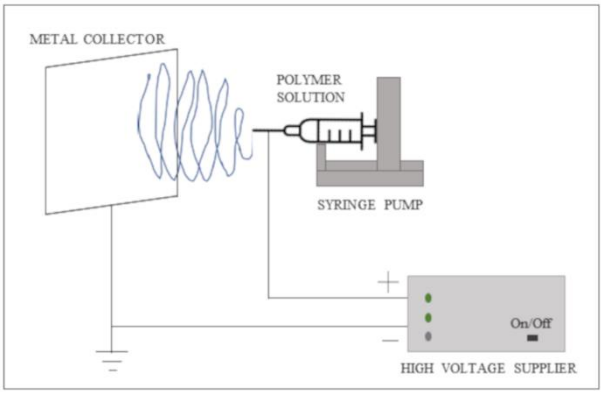

Figure 1. Schematic representation of the electrospinning setup

2.4. Characterization

Shear viscosity of polymer solutions was characterized with a RheolabQC (Anton Paar) rotational rheometer.

The morphology of the electrospun nanofibers was examined by scanning electron microscopy (SEM) using a HITACHI FlexSEM 1000 II microscope. Before SEM analysis, the samples were coated with Al by vapor deposition (NVBJ-300 TH).

The diameter of the nanofibers was determined using Image J software (v. 1.52i, National Institute of Health, USA) from 100 nanofibers as described in the literature (Maleki, Natalello, Pugliese, & Gelain, 2017).

3. Results and Discussion

3.1. PVP/Ethanol System

3.1.1. Effect of PVP concentration

In order in investigate the effect of polymer concentration on the morphology and diameter of nanofibers, solutions of PVP in ethanol of different concentrations varying between 5 wt.% and 14 wt. % were electrospun at a voltage of 17,5 kV, a flow rate of 1.25 mL/h and a tip-to-collector distance of 15 cm.

Figure 2 shows the spread of the electrospun mat on the matellic collector for solution of 5wt.% and 6 wt.% of PVP in ethanol. A white coating can be clearly distinguished showing the deposition of electrospun material on the aluminum foil. However, this pictures do not allow to identify the morphology of the electrospun nanofibers.

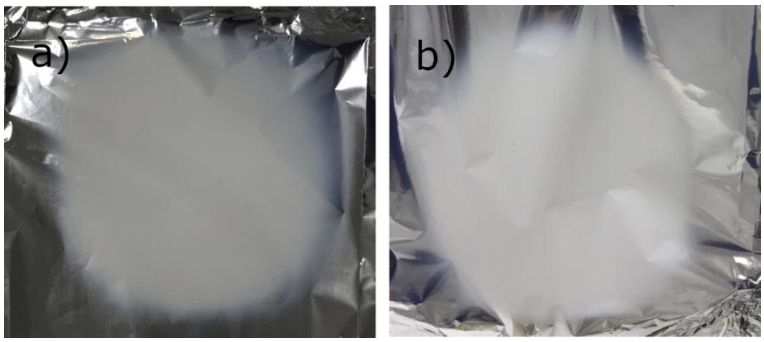

Figure 2. Pictures showing the spread of electrospun nanofiber mats on aluminum foil: a) 5% PVP in ethanol and b) 6% PVP in ethanol.

Therefore, SEM observations were performed. The micrographs obtained for samples with 7 wt.%, 8 wt.% and 10 wt.% of PVP in ethanol corresponding to cases having the most fibrous morphology are presented in Figure 3. Nanofibers diameter distributions are also given for each case. For concentrations lower than 7 wt.%, electrospraying and beaded nanofibers were obtained. According to Figure 3, the most homogenous nanofibers were obtained with a concentration of 7 wt.%. A closer look on SEM micrographs reveals that extra webs are present in both other cases. Moreover, flatter or ribbon like fibers appear for 10 wt. % case. Actually, these results are in correlation with the diameter distributions. A narrower distribution is observed for 7 wt. % which corresponds to the case where the nanofibers are the most homogeneous. When the concentration rises to 8 wt.%, extra webs appear and as it can be expected, the diameter distribution becomes larger. Then, a wider diameter distribution is obtained for 10 wt.5 of PVP in ethanol. Thus, the optimal polymer concentration was identified as 7 wt.% of PVP in ethanol.

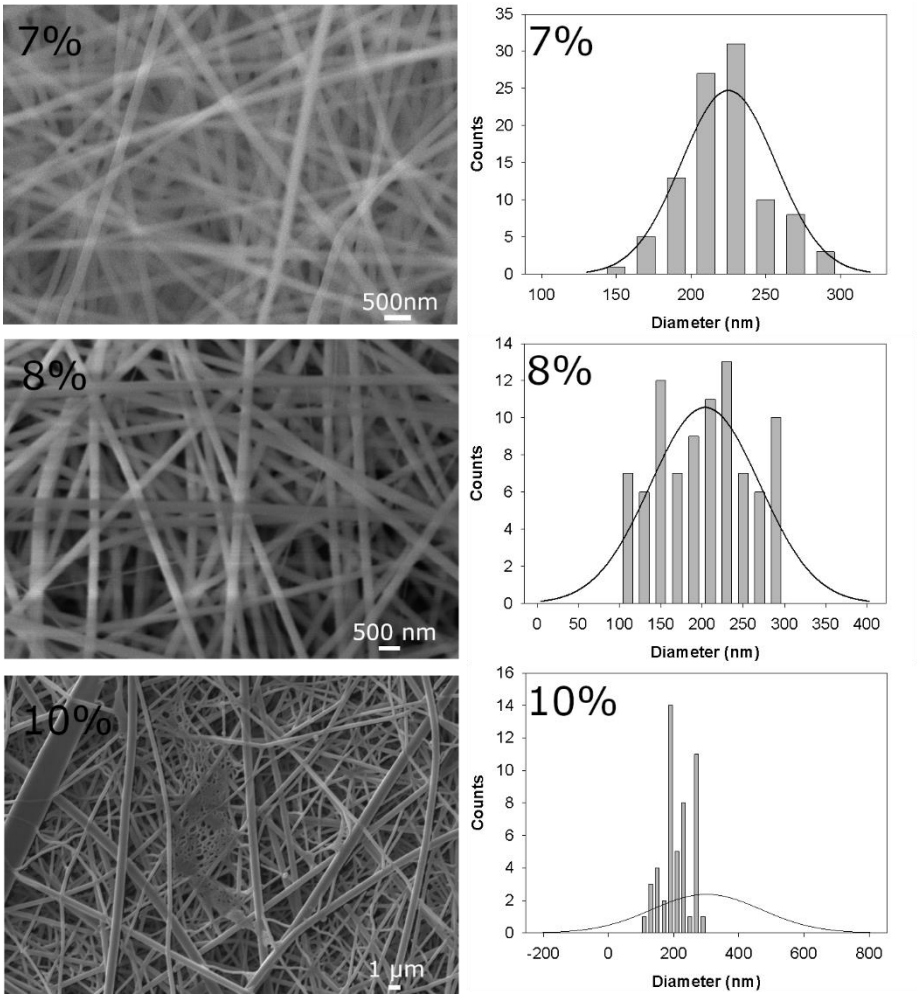

Figure 3. SEM micrographs and corresponding nanofiber diameter distributions of nanofibers obtained for a) 7% PVP in ethanol, b) 8% PVP in ethanol and 10% PVP in ethanol.

The evolution of the mean nanofiber diameter with increasing concentration is presented on Figure 4. Mean diameters around 220 nm and 200 nm were found for 7 wt. % and 8 wt.% of PVP in ethanol, respectively. Then, thicker fibers having a diameter around 300 nm are obtained for 10 wt.%.

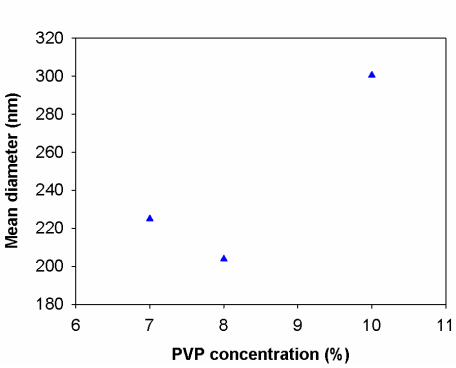

Figure 4. Evolution of the mean diameter of nanofibers with increasing concentration for concentration for which the most homogeneous fibers were observed.

A schematic representation of the evolution of the morphology of nanofibers is presented in Figure 5. For low concentrations, electrospraying occurs and beads are obtained on the collector. When the polymer concentration increases, beaded-nanobibers appear. And then, nanofibers with homogeneous, cylindrical morphology are observed correspondind to the optimum concentration. For higher polymer contents, extra-webs of thinner nanofibers appear. This morphology is followed by the appearance of flat or ribbon-like fibers. The evolution of the morphology is also summarized in Table 1.

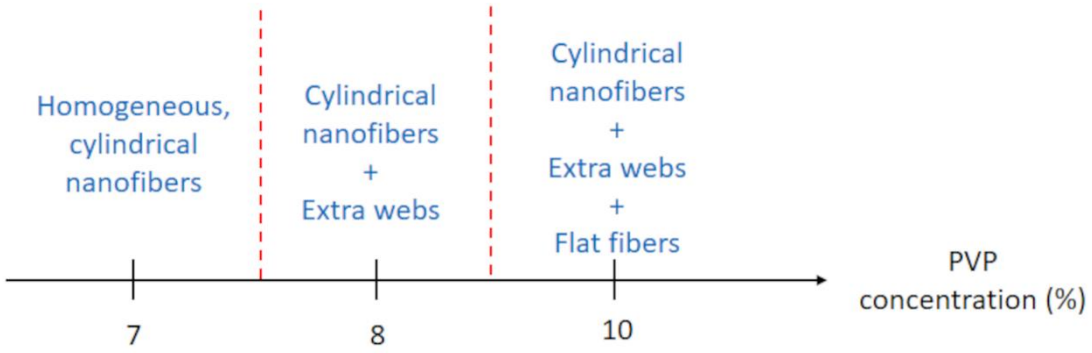

Figure 5. Schematic representation of the evolution of the morphology of nanofibers with increasing PVP concentration.

Table 1. PVP fiber morphology and splaying behavior of polymer solution according to different concentrations

Concentration by weight	Morphology
5	Spray formation
6	Spray formation
7	Uniform continuous fiber
8	Continuous but not uniform fibers
10	Network Structure and non uniform

3.1.2. Effect of voltage

In order to investigate the effect of the voltage, the electrospinning of the different PVP in ethanol solutions was performed at different voltages higer or lower than 17.5 kV without varying the other parameters. For all cases, nanofiber formation was not observed.

3.1.3. Effect of flow rate

The effect of the flow rate was also examined. The experiments were realized with the solution of 7 wt.% of PVP in ethanol which was identified as the optimum concentration. The voltage was maintained at 17.5 kV and the tip-to-collector was kept at 15 cm. The

main results are illustrated in Table 2. According to these results, at a flow rate of 1.00 mL/h, electrospraying is observed and only beads are formed on the collector. On the contrary, at a flow rate of 1.25 mL/h, homogeneous nanofibers are formed. However, similarly to the first case, at a flow rate of 1.50 mL/h, there is no fiber formation and only beads are observed.

Table 2. Electrospinning of 8 wt % PVP/ Ethanol solution with different flow rates.

Distance(cm)	Flow Rate(mL/h)	Voltage(kV)	Observation
15	1.00	17,5	Electrospraying / droplets/beads
15	1.25	17,5	Homogeneous fibers
15	1.50	17,5	Electrospraying / droplets/beads

3.1.4. Shear viscosity of the PVP/Ethanol solution

The evolution of the shear viscosity of PVP in ethanol solutions with increasing shear rate is presented on Figure 6. As expected, the viscosity increases with the increase of PVP concentration. The change in viscosity occurs due to the high molecular weight of the PVP used in this study and the increase in polymer chains as the concentration increases.

In the electrospinning process, fiber formation occurs by stretching the charged jet. The stretching of the jet results in changes on the fiber morphology and diameter with the different concentrations and viscosity of the polymer solution. Viscosity and concentration are two related parameters. For a solution with a very low concentration that is, low viscosity, when the electric field is applied, it is possible to form beaded like morphologies with nanofibers. In other words, electrospray will occur instead of fiber formation with low viscosity. If the concentration starts to increase, the surface tension will be exceeded and bead free homogeneous fibers are formed. However, if the concentration continues to increase and goes beyond a critical value, the fiber morphology changes again and helix-shaped micro ribbons will be observed, also causing the needle tip to clog. Therefore, optimum value should be provided for homogeneous fiber morphology (Unnithan, R.S., & Kim, 2015); (Haider, Haider, & Kang, 2018); (Yang, et al., 2004); (Haide, et al., 2013). It should be noticed that as mentioned in the section on the effect of polymer concentration, the diameter of the nanofibers increases with increasing polymer concentration. The same relationship is also valid with the viscosity.

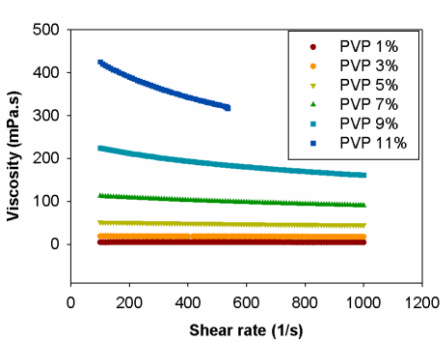

Figure 6. Shear Viscosity vs Shear Rate Graph for PVP/Ethanol Solutions

3.2. PVP/DMSO System

To our knowledge, no study exist on the electrospinning of high molecular weight PVP in DMSO solution in the literature. Moreover, very few studies exist on the electrospinning of PVP in DMSO solutions. Wortmann et al. (Wortmann, et al., 2019) worked on the electrospinning of low molecular weight PVP in DMSO. The authors obtained beaded fibers with beads was obtained from the PVP/DMSO solution formed by needleless electrospinning method.

In this study, high molecular weight PVP in DMSO solutions were prepared and tested. Table 3 presents the different process conditions tested with solution concentration of 8 wt. % at a tip-to-collector distance of 15 cm. Actually, in all cases, a wet coating is formed on the surface of the aluminum foil and no solid fiber mat was observed. This wetness is probably due to the fact that the solvent can not evoparate until the jet arrives on the collector. The pictures of the of wet deposition on aluminum foil after electrospinning of the solution of 8 wt% PVP in DMSO at a voltage of 15 kV, a flow rate of 0.75 mL/h and a tip-to-collector of 15 cm is given in Figure 7.

Table 3. Electrospinning of 8 wt% PVP/DMSO solution with different process conditions

Flow Rate(mL/h)	0.5	0.5	0.5	0.6	0.75	0.75	1.25	1.25	1.25	1.25	1.75	1.75	1.75
Voltage(kV)	15	17.5	20	15	15	17.5	15	17.5	20	22.5	15	17.5	20

Figure 7. Picture of wet deposition on aluminum foil after electrospinning of the solution of 8 wt% PVP in DMSO at a voltage of 15 kV, a flow rate of 0.75 mL/h and a tip-to-collector of 15 cm.

3.3. PVP/DMSO/Acetone System

According to the literature, acetone and DMSO are miscible solvents due to the similarity in their polarity (Wortmann, et al., 2019). Besides, PVP is a polymer soluble in both solvent and therefore, it could be dissolved in a binary mixture of these solvents.

Wortmann et al. (Wortmann, et al., 2019) used needleness electrospinning at very high voltages between 60 and 80 kV to electrospun PVP/DMSO/acetone solutions with DMSO:acetone ratios of 9:1 and 8:2. They obtained porous and bead-free fiber morphology. The authors attributed these result to the volatility and vapor pressure differences of solvents. In this study, in order to increase the volatility of DMSO, PVP solutions in DMSO/acetone binary solvent system were prepared in a 9:1 ratio. However, the fiber mat deposited on the metallic collector remains wet and formed droplets. A picture of wetness observed on the aluminum foil after the electrospinning of a 8 wt. % PVP in DMSO/acetone solution (DMSO/acetone ratio of 9:1) is given as illustration in Figure 8. These results are contrary to the observations of Wortmann et al. which is probably due to the high molecular weight of PVP used in this study but also to the ambient temperature during experiments.

Figure 8. Picture of wet deposition on aluminum foil after electrospinning of the solution of 8 wt% PVP in DMSO/acetone binary solvent system.

3.4. PVP/DMSO/Ethanol System

The other binary solvent system tested for improving the evaporation of DMSO, is DMSO/ethanol system at a ratio of 8:2. PVP solutions were prepared and electrospun. However, the wetness was again observed on the aluminum foil for each case. A picture of wetness observed on the aluminum foil after the electrospinning of a 8 wt. % PVP in DMSO/ethanol solution (DMSO/ethanol ratio of 8:2) is given as illustration in Figure 9.

Figure 9. Picture of wet deposition on aluminum foil after electrospinning of the solution of 8 wt% PVP in DMSO/ethanol binary solvent system.

4. Conclusion

The aim of this study is to investigate the electrospinning behavior of PVP with lower toxicity solvents. Ethanol, DMSO, DMSO/acetone binary sistem and DMSO/ethanol binary systems were used as solvents. Solutions at different concentrations of PVP were prepared and electrospun at various processing conditions. Homogeneous continuous nanofibers with a man diameter around

200nm were obtained when ethanol is used as solvent for 7 wt.% of PVP. The optimum processing conditions were identified as a voltage of 17.5 kV, a tip-to-collector distance of 15 cm and a flow rate of 1.25 mL/h.

Although the solvent evaporates during electrospinning, solvents with lower toxicity such as DMSO are preferable for biomedical applications. Therefore, solutions of PVP in DMSO were electrospun and the results show that DMSO do not evaporate during the trajectory of the jet through the collector.

In order to overcome this wetness problem, DMSO/acetone and DMSO/ethanol binary solvent systems were used to prepare PVP solutions. Despite the solvent system and the change in solvent ratios, the wetness on the collector surface was still present and the solvent was not volatile enough.

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