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Fabrication of Polyvinylpyrrolidone Nanofibers with Green Solvents

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Abstract: In this study, biocompatible Polyvinylpyrrolidone (PVP) nanofibers were produced with ultra-pure water, rose water, lavender water, ethanol, acetone and acetic acid with green electrospinning approach. Polymer solutions were characterized with conductivity, surface tension and viscosity measurements. Morphological analyzes were carried out with Scanning Electron Microscope (SEM). Conductivity, surface tension and viscosity results of PVP/ ultra-pure water, rose water and lavender water solutions were similar. On the other hand, PVP/acetic acid solution has the highest viscosity and lowest conductivity values and PVP/ethanol solution has got the lowest surface tension. In addition; the lowest average fiber diameters were obtained from ultra-pure water, rose water and lavender water solvents but there are some beads on the nanofiber structure. The smoothest nanofibers without beads were obtained from PVP/ethanol but it was observed that average fiber diameter is about 724 nm higher than other PVP solutions. Therefore, ethanol was chosen as a co-solvent to enhance fiber morphology for second part of study. Moreover; the relation between solution conductivity, nanofiber diameter increases and fiber diameter were determined and it was found that nanofibrous surface diameter increases and fiber diameter decreases with the increase of solution conductivity.

Key words: Polyvinylpyrrolidone, Green Solvent, Rose water, Lavender water, Green electrospinning, Nanofiber.

Çevreci Çözücüler ile Polivinilpirolidon Nanolif Üretimi

Özet: Bu çalışmada, çevreci elektro lif çekimi yaklaşımı ile ultra saf su, gül suyu, lavanta suyu, etanol, asetik asit ve aseton gibi farklı çevreci çözücüler ile biyouyumlu polivinilpirolidon (PVP) nanoliflerin üretiminin gerçekleştirilmiştir. Polimer çözeltiler iletkenlik, yüzey gerilimi ve viskozite ölçümleri ile karakterize edilmiştir. Morfolojik analizler Taramalı Elektron Mikroskopu (SEM) ile gerçekleştirilmiştir. PVP/ultra saf su, gül suyu ve lavanta suyu çözeltilerinin iletkenlik, yüzey gerilimi ve viskozite sonuçları; benzer iken PVP/ asetik asit çözeltisi en yüksek viskoziteye ve en düşük iletkenlik değerlerine sahiptir. Diğer taraftan PVP/etanol çözeltisi en düşük yüzey gerilimine sahiptir. En düşük ortalama lif çapı; ultra saf su, gül suyu ve lavanta suyu ile elde edilmiştir fakat boncuklu lifler gözlenmiştir. En düzgün nanolifler PVP/etanol çözeltisinden elde edilirken, ortalama lif çapının 724 nm civarında, diğer PVP çözeltilerden daha kalın olduğu gözlenmiştir. Bu nedenle çalışmanın diğer kısmında lif morfolojisini geliştirmek için etanol yardımcı çözücü olarak seçilmiştir. Ayrıca, çözelti iletkenliği, lif çapı ve nanolifli yüzey çapı arasında ilişki olduğu tespit edilmiş ve iletkenlik arttıkça, lif çapının azaldığı, nanolifli yüzey çapının ise arttığı belirlenmiştir.

Anahtar kelimeler: Polivinilpirolidon, Çevreci çözücü, Gül suyu, Lavanta suyu, Çevreci elektro lif çekim yöntemi, Nanolif

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1. Introduction

Clean and safe production has gained importance with the increasing industrial pollution. Green electrospinning approach is important in terms of environmental friendly nanofiber production and is necessary especially for some application areas such as medical, food etc. [1, 2]. An important part of the green electrospinning method is using green solvents. Briggs and Arinzeh [3] reported 12 criteria that green solvents should fulfil related to availability, price, recyclability, grade, synthesis, toxicity, biodegradability, performance, stability, flammability, storage and renewability. Researchers are considered of this report to choose suitable solvent for their studies. In this study, ultra-pure water, rose water, lavender water, ethanol, acetone and acetic acid were used as green solvents and these solvents were selected from solvent selection guides [4].

Electrospinning is one of the most common method for the production of polymeric nanofibers. Nanofibers have unique properties such as large specific surface area (m^2/g) , high porosity, small and controllable pore size and very small fiber diameter etc. [5-7]. But there are also some disadvantages of electrospinning in terms of green production (unsuitable polymers, hazardous solvents etc.). The most important disadvantage of this method is using of harmful, toxic and harsh solvents (chloroform, dichloromethane etc). These organic solvents are used for insoluble polymers in distilled water or green solvents. Drawbacks of organic solvents are expensive, toxic and non-ecofriendly. Also, organic solvents are not preferable for certain applications such as tissue engineering, wound dressing, drug delivery [8-10]. It is well known from the literature [11]; solution properties (solvent type, conductivity, viscosity etc.) effect spinnability and nanofiber morphology significantly.

There are numerous studies in the literature about effects of solvents on the electrospinning and nanofiber morphology. Cay et al. [12] have investigated effects of solvent mixtures on the morphology of thermoplastic polyurethane (TPU) nanofibers and N.N-dimethylformamide (DMF), tetrahydrofuran (THF) and ethylacetate (EA) were used as solvents. They determined that fiber diameter increased with THF volume fraction. And diluting TPU solutions with 10 or 20% of EA has positive effect on the fiber diameter. Casasola et al. [13] have tested effects of acetone (AC), 1,4-dioxane tetrahydrofuran (THF), dichloromethane (DCM), chloroform (CHL). (DX). dimethylformamide (DMF) and dimethylacetamide (DMAc) on the electrospun polylactic acid (PLA) fibers morphology, productivity and diameter distribution. They reported that solvent properties such as boiling point, viscosity, conductivity and surface tension, have a significant effect on the spinnability, fiber morphology and fiber diameter distribution. In 2008, Veleirinho et al [14] investigated about solvent and polymer concentration effects on the poly (ethylene terephthalate) nanofibers. They used trifluoroacetic acid/dichloromethane (TFA/DCM) as a solvent various mixture rate such as 30/70, 50/50, 70/30 and 100/0, respectively. They determined that solvent properties effect fiber morphology especially diameter. In addition, it was found that fiber diameter and surface tension decrease with increasing TFA rate in the solvent mixture.

In this study, polyvinylpyrrolidone (PVP) was used as the raw material because of specific advantages such as biocompatible, soluble with many solvents, non-toxic and hydrophilic. PVP nanofiber-based materials have important biomedical application areas, for instance in medicine [15, 16], drug delivery and release systems [17-22],

biomedical [23], and bioactive packaging applications [24]. In literature; there are some studies about effects of solvents on PVP nanofiber morphology. Chuangchote et al. [25], investigated effects of solvents on electrospinnability for electrospinning of PVP. They used seven solvents such as; methanol, ethanol, 2-propanol, 1,2-dichloroethane, water, chloroform and dichloromethane. They reported that methanol is the most suitable solvent for the electrospinning of PVP. Yang et al. [26] studied about PVP nanofiber optimization with different solvents such as; N,N-dimethylformamide (DMF), ethanol and dichloromethane (DMC). They obtained very small nanofibers (20 nm) and ethanol/DMF (50/50) mixture was found an optimum solvent to produce PVP nanofibers.

As mentioned above; green production, green chemistry and green solvents come into prominence last decades significantly. Rose and lavender water which were used as solvents in this study are local products in Turkey, Isparta. As well known; Turkey, Isparta is the most important rose (*Rosa damascena* Mill.) supplier in the world. Rose and lavender water which can be used for some important application areas such as soaps, food flavoring, cosmetics and perfumes because of its natural anti-oxidant activity and pleasant odor [27]. In literature there is no study about rose water and lavender water usage as a solvent for electrospinning. The most important difference of this study from literature is green electrospinning approach and determination of the most suitable green solvent selection for PVP nanofibers.

The aim of this study is production and determination of most suitable green solvent or solvent mixture for electrospinning of PVP nanofibers.

2. Material and Method

2.1 Material

In this study, PVP (Mw 360.000 g/mol) was used as a polymer (Figure 1), ultra-pure water, rose water, lavender water, ethanol, acetone and acetic acid were used as a solvent. PVP, ethanol, acetone and acetic acid were purchased from Sigma Aldrich Company, rose water was supplied from Rosense brand (Gülbirlik) in Isparta in Turkey, lavender water was supplied from faculty of agriculture at Suleyman Demirel University. Ultra-pure water was obtained from Millipore Milli-Q System with conductivity of 18.0 M Ω .cm.



Figure 1. Chemical structure of PVP [26]

There are two parts in this study such as; pure (Part I) and binary (Part II) solvent system. Firstly; PVP polymer solutions were prepared with different green solvents (Table 1). Second part of this study, PVP polymer solutions were prepared with solvent mixtures using ethanol (Table 2). All the PVP solutions were prepared under the same conditions such as stirring time, stirring speed (rpm), temperature etc.

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Sample Codes	Polymer Concentration (%)	Solvent
PVP1	12	Ethanol
PVP2	12	Ultra-pure water
PVP3	12	Rose water
PVP4	12	Lavender water
PVP5	12	Acetic acid
PVP6	12	Acetone

 Table 1. PVP sample codes with various green solvents (Part I).

 Table 2. PVP sample codes with various green solvent mixtures using ethanol (Part II).

Sample Codes	Polymer Concentration (%)	Solvent Mixture (50/50)
PVP1.2	12	Ultra-pure water/Ethanol
PVP1.3	12	Rose water/ Ethanol
PVP1.4	12	Lavender water/Ethanol
PVP1.5	12	Acetic acid/ Ethanol
PVP1.6	12	Acetone/ Ethanol

2.2 Method

Conductivity, surface tension and viscosity measurements all of the PVP solutions were measured by conductivity meter Selecta CD 2005, Biolin Scientific Sigma 702 with Wilhelmy Plate method Lamy Rheology, B-One Touch Screen viscometer under 5 s⁻¹ shear rate, respectively.

Conventional needle electrospinning apparatus was used production of nanofiber webs. Schematic description of electrospinning apparatus is given in Figure 2.



Figure 2. Schematic representation of the electrospinning apparatus [28]

Polymer solutions were prepared and fed by a syringe pump under constant rate. High voltage was applied for electrostatic area between the needle tip and collector which causes jet formation and breakage to the thinner jets. During the electrospinning process solvent evaporates and dry nanofibers are randomly collected on the aluminum foil (Figure 3).



Figure 3. Images of the electrospinning apparatus

During the spinning process, 26.4 kV voltages, 0.8 mL/h solution feed rate, and 19.0 cm distance between the electrodes were applied for all solutions. Spinning experiments were achieved under the ambient conditions as 35 ± 1 % humidity and 20.5 ± 1 °C degrees (Table 3).

Table 3. Process parameters of the electrospinning				
Voltage Distance between Feed rate Humidity Temperatu				Temperature
(kV)	electrodes (cm)	(mL/h)	(%)	(°C)
26.4	19.0	0.8	35	20.5

Nanofibrous surface morphology (bead presence, fiber diameter etc.) was analyzed with the Scanning Electron Microscope (SEM) FEI Quanta 250 FEG model. Then, the average fiber diameter was measured using 100 different fiber diameters for each sample from the SEM images using the Image J software. The fiber diameter uniformity coefficient was counted up with a method that uses the same principle as for molar mass distribution in chemistry science [29]. The number average and weight average values were calculated using equation (1) and (2) given below.

$$A_{n} = \frac{\sum n_{i}d_{i}}{\sum n_{i}} \text{(average number)}$$

$$A_{w} = \frac{\sum n_{i}d_{i}^{2}}{\sum n_{i}d_{i}} \text{(average weight)}$$
(1)
(2)

d_i: fiber diameter n_i: fiber number

The fiber uniformity coefficient was determined by ratio A_w/A_n . An ideal optimum value should be close to 1 for uniform fibers [29].

3. Results and Discussion

3.1 Solution Properties Results

Firstly; solution properties such as conductivity, surface tension and viscosity were determined. All solution properties results are given in Table 4 and Table 5.

Sample	Conductivity	Surface Tension	Viscosity (Pa.s)	
Codes	(µS/cm)	(mN/m)	(shear rate 5 ⁻¹)	
PVP1	8.61	19.42	0.456	
PVP2	86.5	54.08	0.739	
PVP3	78.3	53.85	0.777	
PVP4	87.1	47.84	0.729	
PVP5	2.28	26.77	2.273	

Table 4. Conductivity, surface tension and viscosity results of Part I PVP solutions

According to the Table 4; viscosity and conductivity values of PVP solutions with ultrapure water (PVP2), rose water (PVP3) and lavender water (PVP4) were too close to each other. PVP1 and PVP5 samples has similar conductivity values relatively PVP5 has the lowest conductivity (2.28 μ S/cm). It is determined that these results are also compatible with pure solvent conductivity values without PVP polymer conductivity properties. On the other hand, PVP solution with ethanol (PVP1) has got the lowest and with acetic acid (PVP5) has got the highest viscosity values. According to Table 4, PVP2 and PVP3 surface tension results are very close to each other and PVP1 has the lowest surface tension 19.42 mN/m for Part I of the study solution of PVP6 (with acetone) could not prepared because solution turned into gum.

Table 5. Conductivity, surface tension and viscosity results of Part II PVP solutions

Sample	Conductivity	Surface Tension	Viscosity (Pa.s)
Codes	(µS/cm)	(mN/m)	(shear rate 5 ⁻¹)
PVP1.2	22.8	16.90	1.023
PVP1.3	20.7	26.12	1.022
PVP1.4	21.4	26.34	1.017
PVP1.5	9.82	22.45	0.955
PVP1.6	14.73	18.51	0.170

PVP1.2, PVP1.3 and PVP 1.4 viscosity and conductivity values are too close with each other. But, conductivity values of PVP1.2, PVP1.3 and PVP 1.4 lower than PVP2, PVP3 and PVP4 because, conductivity of pure ethanol (0.04 μ S/cm) value is lower than ultra-pure water (20.16 μ S/cm), rose water (17.80 μ S/cm) and lavender water (25.4 μ S/cm). However, sample of PVP1.2, PVP1.3 and PVP 1.4 viscosity values higher than PVP2, PVP3 and PVP4. Similarly, ethanol addition decreases PVP1.5 viscosity from 2.273 to 0.955 dramatically. Also, conductivity of PVP1.5 is higher than PVP5 and PVP1. It was thought that ethanol increased solution conductivity with acetic acid. In addition, PVP6 (PVP/acetone) could not prepared but, PVP1.6 can be prepared with ethanol. It was understood that ethanol was enriched spinnability for PVP polymer. Ethanol addition decrease surface tension values significantly. the lowest surface tension was determined 16.90 mN/m with PVP1.2 (PVP/ethanol: ultra-pure water) for Part II of the study.

3.2 Fiber Morphology Results

SEM images and fiber diameter histograms of PVP nanofibers from Part I that include various solvents are given in Figure 4 and Figure 5.







PVP2 (PVP/ultra-pure water)







PVP4 (PVP/lavender water)



PVP5 (PVP/acetic acid)

Figure 4. SEM images $(1.000 \times -15.000 \times)$ of PVP nanofiber samples produced with various solvents and fiber diameter histograms



Figure 5. PVP nanofibers fiber diameter and diameter uniformity coefficient results with various solvents

As it has seen from SEM images and histograms; fiber morphology has smooth surface and no beads with ethanol (PVP1) however high average fiber diameter was about 724 nm. Also, there is no bead and membranous structure with acetic acid (PVP5) while it was observed that very coarse fibers and it can be said microfiber because of higher than 1000 nm average fiber diameter. Relatively lower average fiber diameters were obtained using PVP/ultra-pure water, rose water and lavender water (PVP2, PVP3 and PVP4) solutions. Generally, it is possible to say that the lowest average fiber diameters were obtained from ultra-pure water (161.44 nm), rose water (157.04 nm) and lavender water (125.57 nm) solvents but there are some beads on the nanofiber structure which is undesirable defect. PVP/ethanol is the smoothest nanofiber without beads but average fiber diameter is coarser (724.47 nm). The main objective of this study is determining an ideal nanoweb structure with very fine and uniform fibers without beads. Therefore, the most suitable solvent should be identified in terms of these parameters.

When the fiber diameter uniformity results were analyzed, generally uniform nanofibers were obtained from PVP2, PVP3, PVP4 and PVP5. The lowest uniformity was obtained from PVP1 (PVP/ethanol) is 1,521 and the most uniform nanofibers were obtained from PVP5 (PVP/acetic acid) and PVP4 (PVP/lavender water) are 1.007 and 1.049, respectively. Also, there is a unimodal curve for sample of PVP2, PVP3, PVP4 and PVP5 on histograms. Especially PVP5 histogram curve is unimodal and narrow range.

SEM pictures and fiber diameter histograms of PVP nanofibers from Part II that include ethanol and various solution mixtures (50:50) are given in Figure 6 and Figure 7.









Figure 6. SEM images $(1.000 \times -15.000 \times)$ of PVP nanofiber samples produced with ethanol and various solution mixtures (50:50) and nanofiber histograms



Figure 7. PVP nanofibers fiber diameter and diameter uniformity coefficient results with solvent mixtures

As it has known from literature; fiber morphology can be enhanced with three methods at Part II. These methods are increasement of polymer molecular weight, higher polymer concentration and addition of co-solvent to the solvent system [30]. Addition of co-solvent to the solvent system was chosen to improve fiber morphology because spinnability increased. It is clearly seen from Figure 5 and Figure 6, ethanol addition in the solvent system has a positive contribution such as improves fiber morphology significantly without bead and stickiness except the increase of the fiber diameter. Because of fiber morphology improvement, at the second part of the study ethanol was used as a co-solvent with all other solvents by the ration of 50/50. It was possible to produce smoother and beadless nanofibers in the second part of this study. As it could be clearly seen from histograms, sample of PVP1.2, PVP1.3, PVP1.4 and PVP1.6 has got unimodal curve. Fiber morphology was influenced positively with addition of

ethanol in the solvent system for PVP1.2, PVP1.3 and PVP1.4. On the other hand, average fiber diameter was increased and solution conductivity values were decreased with addition of ethanol in the solvent system. It is well known in the literature, there is a relationship between fiber diameter and solution conductivity. When solution conductivity is increased, it can be obtained finer fiber diameter and beadless fiber morphology [31]. Our results are compatible with literature except beadless fiber morphology. But beads can be eliminated increasing polymer concentration for sample of PVP2, PVP3 and PVP4. In addition, the most uniform, finest nanofibers with 469.97 nm and the smoothest nanofibers were obtained with lavender water/ethanol solvent system in the second part of this study. The most uniform nanofibers were obtained from ultra-pure water and lavender water solutions are 1.018 and 1.020, respectively. Also, sample of PVP1.5 (PVP/ethanol/acetic acid) was affected from the addition of ethanol such as fiber diameter and fiber morphology. For instance; sticky and membranous structure was removed and average fiber diameter was decreased from 1170.9 nm to 609.1 nm with addition of ethanol. Moreover, viscosity values decreased from 2.273 Pa.s to 0.955 Pa.s and solution conductivity increased from 2.28 µS/cm to 9.82 µS/cm. Besides, sample of PVP5 and PVP1.5 fiber diameter and fiber morphology analyses were expected results in terms of literature. According to the literature; average fiber diameter increases with viscosity and decreases with solution conductivity [33]. As it has been seen from histogram curve of PVP1.5 (PVP/Ethanol/Acetic acid), the lowest uniformity coefficient was obtained from this sample is 1.348. Lastly, during the solution preparation studies, it was observed that PVP/acetone is not possible to electrospin because of solution gumming. In Addition, ethanol to acetone solvent (50:50) improves both solution properties and spinnability dramatically.

Another result of this study is the relation between solution conductivity and nanoweb diameter which is given in Figure 8.



Figure 8. Relationship between solution conductivity and nanoweb diameter

It is clearly seen from Figure 8, there is a strong relation between solution conductivity and nanoweb diameter that nanoweb diameter increases with solution conductivity. On the other hand, it is possible to say that nanoweb diameter can be related with spinning performance which is very important for electrospinning process. According to the Figure 7, PVP4 sample has the highest nanoweb diameter (12.2 cm) and the highest solution conductivity (87.1 μ S/cm) in addition pure lavender water has the highest conductivity value (25.4 μ S/cm), too. On the other hand, solvent conductivity value has effect on this tendency for instance PVP2 (ultra-pure water), PVP3 (rose water) and PVP4 (lavender water) samples solvents have relatively higher conductivity values such as 86.5 μ S/cm, 78.3 μ S/cm and 87.1 μ S/cm, respectively. Therefore, it is again demonstrated that importance of suitable solvent selection for electrospinning.

Similarly, there is a relationship between solution conductivity and average fiber diameter (Figure 9). It is also reported in the literature; fiber diameter decreases with solution conductivity increasement [31, 32]. Therefore, our results are compatible with literature.



Figure 9. Relationship between solution conductivity and average fiber diameter

Another relationship between solution conductivity and fiber diameter uniformity is shown in Figure 10. According to this figure, it is possible to say fiber diameter uniformity values has positive tendency with solution conductivity increasement.



Figure 10. Relationship between solution conductivity and fiber diameter uniformity coefficient

All fiber properties results obtained from this study are summarized in Table 6. PVP1.4 was determined as an optimum sample in terms of fiber diameter, uniformity and morphology results.

Sample Codes	Weight Average Diameter (A _w)(nm)	Number Average Diameter $(A_n)(nm)$	Fiber Diameter Uniformity Coefficient (A_w/A_n)	Nanoweb Diameter (cm)	Nanoweb Morphology
PVP1	1101.8	724.4	1.521	5.2	Smooth
PVP2	178.2	161.4	1.104	11.8	With beads
PVP3	167.9	157.0	1.069	9.8	With beads
PVP4	131.7	125.5	1.049	12.2	With beads
PVP5	1179.1	1170.9	1.007	3.8	Partly sticky
PVP1.2	558.4	548.5	1.018	7.7	Smoothest
PVP1.3	554.6	501.5	1.106	9.6	Smoothest
PVP1.4	479.1	470.0	1.020	6.4	Smoothest
PVP1.5	821.2	609.1	1.348	5.5	Smooth
PVP1.6	809.4	763.7	1.060	6.1	Smooth

Table 6. All the results of fiber properties of PVP samples

4. Conclusion

This study was carried out for the production of PVP nanofibers using various solvents with green electrospinning approach. According to the solution properties results; PVP solutions with ultra-pure water, rose water and lavender water have the similar viscosity, surface tension and conductivity values and these results are also similar with addition of ethanol to these solvents. Fiber morphology results showed that the finest and uniform nanofibers were obtained from PVP samples with ultra-pure water, lavender water and rose water. Because of poor fiber morphology (bead and stickiness defects) of PVP samples with these aqueous solutions; ethanol was used as a co-solvent to improve fiber web morphology. As it is expected, it was observed that ethanol addition improved fiber morphology significantly except increasement of fiber diameter. Generally, smooth and beadless nanofibers were obtained using ethanol as a co-solvent. In addition, pure acetone is not suitable solvent for PVP, but addition of ethanol as a co-solvent was provided to obtain uniform solution and electrospinnability. Moreover, it was obtained that microfibers from PVP/acetic acid and fiber diameter changed from micro to nano scale with ethanol addition. Another important result in this study, solution conductivity relationships with nanoweb diameter and average fiber diameter. Nanoweb diameter increased and average fiber diameter decreased with increasement of solution conductivity. Our findings showed that an ideal nanoweb structure in terms of fiber diameter, diameter uniformity and morphology is PVP/ethanol/lavender water and it is possible to say that this nanoweb material has a potential to use cosmetic, medical application areas in which green production has vital importance.

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