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Araştırma Makalesi

Electrospinning of PVP Nanofibers and Optimization with Taguchi Experimental Design

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Abstract: The aim of this study is the determination of optimum process parameters which will provide the finest and the most uniform electrospun Polyvinylpyrrolidone (PVP) based nanofibers with Taguchi experimental design. For the designed experimental setup, parameters (solvent type, polymer concentration, voltage, distance between the electrodes, solution feed rate and humidity) were used which effect the electospinning process significantly. For this purpose, the appropriate orthogonal array was selected to determine the factors and levels at Taguchi experimental design application. The experimental design aimed which provides to be reduced the number of experiments and minimised the effect of uncontrollable factors with less experiments to obtain target value by using Taguchi orthogonal arrays. In the experimental studies of paper, firstly PVP polymer solutions such as conductivity, surface tension and viscosity were determined with various PVP concentrations (10, 12, 14 wt %) and solvents (ethanol, dimethylformamide, dimethylacetamide, chloroform, acetic acid and distilled water). Scanning Electron Microscope (SEM) images of electrospun PVP based nanofibrous surfaces were obtained, average fiber diameter and fiber diameter coefficient values were calculated by ImageJ image analyses software and fiber diameter distribution histogram curves were obtained by SPSS program. Experimental results were analyzed and commented by Taguchi method in MINITAB program with variance analysis. According to the results; solvent type has the highest effect on the electrospinning of PVP nanofibers. In this study, it is predicted to save in terms of time and cost with decreasing the number of experiments by Taguchi experiment design.

Key words: Polyvinylpyrrolidone, electrospinning, optimization, Taguchi experimental design, nanofiber.

Elektro Lif Çekim Yöntemi ile PVP Nano Lif Üretimi ve Taguchi Deneysel Tasarımı ile Optimizasyonu

Öz: Çalışmanın amacı; Taguchi deneysel tasarımı uygulanarak elektro lif çekimi yöntemi ile en ince ve en üniform Polivinilpirolidon (PVP) esaslı nano liflerin üretimini sağlayacak optimum proses parametrelerinin belirlenmesidir. Tasarlanan deney düzeneğinde elektro lif çekimine önemli ölçüde etki eden parametreler (çözücü çeşidi, polimer konsantrasyonu, voltaj, elektrotlar arası mesafe, çözelti besleme hızı ve nem) kullanılmıştır. Taguchi deney tasarımı uygulamasında, amaca uyan faktör ve seviyeler belirlenerek uygun ortogonal dizin seçilmiştir. Taguchi ortogonal

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dizinleri kullanılarak, hedef değere ulaşmak için yapılan deney sayısı azaltılmış ve daha az deneyle kontrol edilemeyen faktörlerin etkisini de en aza indirgeyen bir deney tasarımı amaçlanmıştır. Yapılan deneysel çalışmalarda öncelikle PVP polimeri kullanılarak farklı konsantrasyonlarda (% 10, 12, 14) hazırlanan çözeltilerin iletkenlik, yüzey gerilimi ve viskozite özellikleri ölçülmüştür. Elektro lif çekimi ile üretilen PVP esaslı nano lifli yüzeylerin Taramalı Elektron Mikroskobu (SEM) görüntüleri alınmış, Image J görüntü analiz programı kullanılarak ortalama lif çapı ve lif çapı üniformite katsayıları hesaplanmış ve SPSS programı ile çap dağılımı histogram eğrileri oluşturulmuştur. Deney sonuçları, varyans analizi kullanılarak MINITAB programında Taguchi yöntemiyle incelenerek yorumlanmıştır. Sonuçlara göre; elektro lif çekimi ile üretilen PVP nanolifler üzerinde çözücü tipi en fazla etkiye sahiptir. Bu çalışmada, Taguchi deneysel tasarımı ile deney sayısının azaltılmasıyla zaman ve maliyet bakımından kazanç sağlanması öngörülmektedir.

Anahtar kelimeler: Polivinilpirolidon, elektro lif çekimi, optimizasyon, Taguchi deney tasarımı, nanolif.

1. Introduction

Nanotechnology is a large interdisciplinary field that can be used in many areas such as filtration, medicine, energy and material science etc. [1-4]. With the development of this technology, new production techniques have emerged in recent years. The products have become multi-functional structures with multiple features [5]. This technology allows working with many polymers such as Polyvinylpyrrolidone (PVP) [6-8]. PVP is a biocompatible polymer and has found a wide place in textile industry, especially in medical textiles and can be produced as a nanofibrous structure by electrospinning method [9-11]. Electrospinning is one of the methods aims increasing the performance of nanotechnology for textile applications. By this method, quite thin nanostructured fibers can be produced and fiber diameter can be controlled [12-13]. The fibers which were obtained with this method are much thinner than the diameter of human hair [14]. There are some parameters which provides high quality of performance in the electrospinning process. Determining the optimum values on the process parameters and obtaining these optimal values are highly long and costly processes. In this respect, production conditions need to be determined quickly.

Different response variables are obtained by changing the input variables in a process. The analysis and interpretation of the different results arising from the applied changes are the subject of the experimental design. It is a method of organizing the effects of the factors on the process efficiently [15-17]. The experimental design idea is an significant strategy which is used in new product design, manufacturing process development and process improvement. Firstly, it is important to determine the factors that influence the production of nanofibers and to determine the appropriate factor levels to control factors [18]. The number of experiments is calculated according to the developed orthogonal arrangements in Taguchi method which reduces the total number of experiments with respect to the factors and levels [19]. In this way, it makes possible to decrease the total cost and the total time required of the experiments [20]. According to Deming, usual (system welded) or special (unusual) reasons can cause variation on the product. In order to improve quality; Taguchi had taken attention the reduction of variation in production processes [21]. Improving the quality of the materials produced is a result of efforts to improve customer satisfaction by minimizing dissatisfaction with the product. Factors affecting quality; market, management, human, motivation, material and equipment [22]. For the purpose of research and development in laboratory conditions, each experiment has several parameters affecting the experiment. It is possible to use the appropriate statistical methods to perform the experiments and to evaluate and analyze the results [23].

In literature; there are several studies about electrospinning of PVP nanofibers [24-27]. Hovewer there are limited studies about electrospinning optimization with Taguchi experimental design. Dong et al., [28] produced Poly (lactic acid) (PLA)/halloysite nanotube (HNT) composite fibers by using electrospinning technique. L9 orthogonal array was selected with four factors of applied voltage, collector distance, feed rate of solution and HNT concentration at three different levels. Albetran et al., [29] was used L9 orthogonal array to investigate titanium isopropoxide concentration within PVP polymer using electrospinning process. Elkasaby et al., [30] has chosen polyvinyl alcohol as a polymer for optimization of electrospinning process. Analysis of variance (ANOVA) and the Taguchi L27 orthogonal array are employed to determine the impact of process control parameters.

In this paper, L18 orthogonal array was selected for the Taguchi experimental design to optimize electrospinning of PVP with several factors (PVP concentration, solvent type, voltage, distance between the electrodes, solution feed rate and chamber humidity). The difference of this study from the literature is L18 mixed factor orthogonal array type and solvent number, chamber humidity factors for the optimization.

2. Material and Method

In this study, PVP (Sigma Aldrich) polymer, which has 360.000 g/mol molecular weight was used as a polymer. Ethanol (ETN) (EMSURE), dimethylformamide (DMF) (Sigma Aldrich), dimethylacetamide (DMAC) (Merck), chloroform (CHL) (EMSURE), acetic acid (AA) and distilled water (DW) were used as the solvents. All the solutions were prepared at 10, 12, 14 wt % PVP concentrations. 18 different PVP samples were prepared according to L18 orthogonal array [31].

Firstly; all solutions were prepared under the same conditions (room temperature, stirring time, etc.). Then, solution properties such as conductivity, surface tension and viscosity were determined. Conductivity and surface tension properties were determined by a conductivity meter (CD-2005) and Wilhelmy method (Biolin Scientific) using a platinum plate and a highly precise electronic balance respectively. In Wilhelmy method, a thin plate is used to measure equilibrium surface or interfacial tension at air-liquid or liquid-liquid interfaces [32]. PVP solution viscosity was measured using a Lamy Rheology at 25 °C with 5 s^{-1} shear rate.

The experimental setup of electrospinning is shown in Figure 1. It consists of a syringe pump filled with a polymer solution, which is connected into high voltage area. The fibers were collected on a collector which has certain distance from the syringe tip. During the transition of the polymer solution into electrical fields, Taylor cone and jets are observed thanks to high voltage.

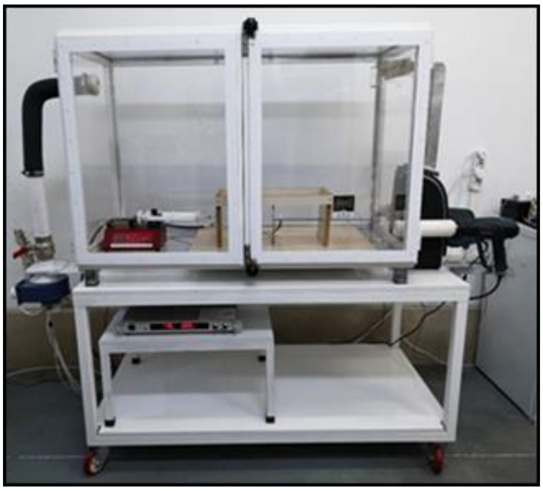

Figure 1. Electrospinning system used for experimental studies

During the spinning process, constant conditions were applied as can be seen in Table 1.

Table 1. L18 orthogonal array of electrospinning parameters [33].

	Parameters					
Run	Solvent type	Poly. Conc. (%)	Voltage (kV)	Distance (cm)	Feed rate (ml/sa)	Humidity (%)
1		10	24.4	17	0.7	25
2	ETN	12	26.4	19	0.8	30
3		14	28.4	21	0.9	35
4		10	24.4	19	0.8	35
5	AA	12	26.4	21	0.9	25
6		14	28.4	17	0.7	30
7		10	26.4	17	0.9	30
8	DMF	12	28.4	19	0.7	35
9		14	24.4	21	0.8	25
10		10	28.4	21	0.8	30
11	CHL	12	24.4	17	0.9	35
12		14	26.4	19	0.7	25
13		10	26.4	21	0.7	35
14	DMAC	12	28.4	17	0.8	25
15		14	24.4	19	0.9	30
16		10	28.4	19	0.9	25
17	DW	12	24.4	21	0.7	30
18		14	26.4	17	0.8	35

For this study, Taguchi experimental design was used to procude all 6 factors, mixed level (L18) orthogonal arrays in order to provide an effective approach for optimizing the fiber diameter.

Firstly, nanofibrous membranes were taken by scanning electron microscopy (SEM) FEI Quanta 250 FEG model under 1.000 and 20.000x magnifications. To determine the average fiber diameter (nm), fiber diameter uniformity coefficient and the nanofibrous surface morphology, Image J software was used by taking 100 different diameter values on the SEM image for each sample.

Then, fiber diameter uniformity coefficient values were calculated using the method which was given in below (1 and 2). This method has the same principle as for molar mass distribution in chemistry science [34]. The number average and weight average values were calculated using equations (1) and (2) given below:

d_i = fiber diameter
n_i = fiber number

$$A_n = \frac{\sum n_i d_i}{\sum n_i} \quad \text{(number average)} \tag{1}$$

$$A_w = \frac{\sum n_i d_i^{\,2}}{\sum n_i d_i} \quad \text{(weight average)} \tag{2}$$

The fiber diameter uniformity coefficient (FDUC) was determined using equation 3; the optimum value should be very close to 1 for uniform fibers [33].

$$\mathrm{FDUC} = \frac{A_w}{A_n} \quad (3)$$

3.Results and Discussion

3.1 Solution properties results

Solution properties results such as conductivity, viscosity and surface tension with various PVP concentrations and solvents were given in Figure 2, 3 and 4. According to the Figure 2, solution conductivity shows significant difference between the solvent types. It was observed that the highest conductivity values were obtained with water solvent while the lowest was measured with chloroform. In addition, conductivity increases as the PVP polymer concentration increasement with DMF and water solvents.

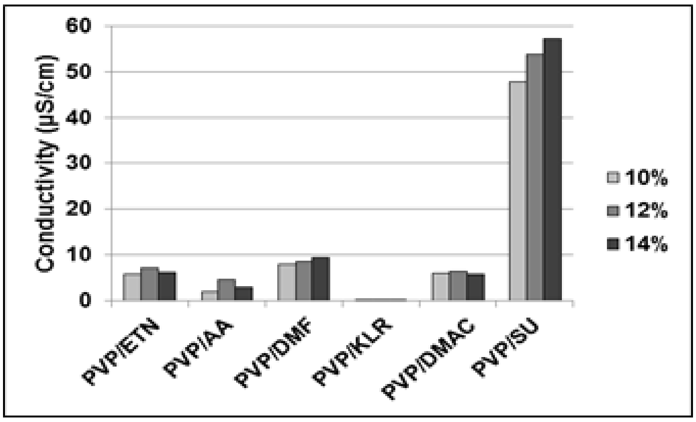

Figure 2. Conductivity results of PVP solutions with various solvents and concentrations.

Figure 3 shows the surface tension values of PVP solutions with various PVP concentrations and solvents. The highest surface tension values were obtained with water solvent while the lowest with chloroform. On the other hand, PVP polymer concentration has no effect on the surface tension except PVP/water. It was observed that surface tension decreases with PVP concentration for PVP/water solution.

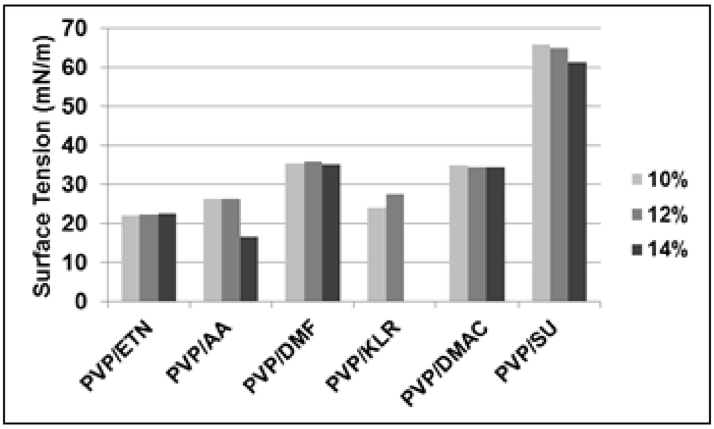

Figure 3. Surface tension results of PVP solutions with various solvents and concentrations.

Viscosity measurement results of PVP solutions with various solvents and concentrations are seen in Figure 4. It is clearly seen that the highest viscosity (2011 mPa) was obtained from PVP/acetic acid solution at 14 wt % PVP while the lowest with PVP/CHL. It is possible to say that, generally solution viscosity increases with PVP concentration increasement.

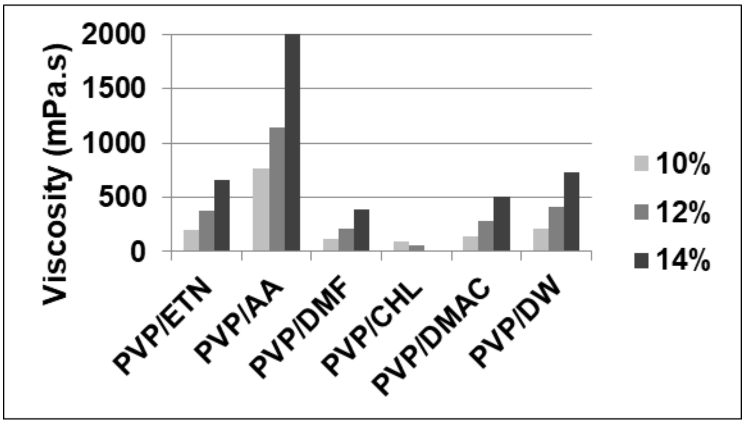

Figure 4. Viscosity results of PVP solutions with various solvents and concentrations.

3.1 Fiber morphology results

The six parameters such as solvent type, polymer concentration, voltage, distance between the electrodes, solution feed rate and chamber humidity were chosen for the Taguchi experimental study. Experimental results of fiber diameter and fiber diameter uniformity coefficient using L18OA are given in Table 2.

Table 2. Experimental results of fiber diameter and fiber diameter uniformity coefficient using L18OA [33]

Parameters							**Average Fiber Diameter (nm)**	**Fiber diameter uniformity coefficient**
Run	**Solvent type**	**Polymer conc. (%)**	**Voltage (kV)**	**Distance (cm)**	**Feed rate (ml/h)**	**Humidity (%)**		
1		10	24.4	17	0.7	25	443.5	1.018
2	ETN	12	26.4	19	0.8	30	688.6	1.016
3		14	28.4	21	0.9	35	948.1	1.040
4		10	24.4	19	0.8	35	802.5	1.026
5	AA	12	26.4	21	0.9	25	1257.4	1.035
6		14	28.4	17	0.7	30	1320.5	1.011
7		10	26.4	17	0.9	30	108.0	1.078
8	DMF	12	28.4	19	0.7	35	129.1	1.087
9		14	24.4	21	0.8	25	234.0	1.098
10		10	28.4	21	0.8	30	1596.8	1.134
11	CHL	12	24.4	17	0.9	35	1865.9	1.132
12		14	26.4	19	0.7	25	2588.1	1.040
13		10	26.4	21	0.7	35	61.1	1.062
14	DMAC	12	28.4	17	0.8	25	96.5	1.164
15		14	24.4	19	0.9	30	128.5	1.098
16		10	28.4	19	0.9	25	79.0	1.087
17	DW	12	24.4	21	0.7	30	115.2	1.130
18		14	26.4	17	0.8	35	178.9	1.212

The SEM images and histograms of the nanofibers produced by the first 3 experiments (10-12-14 wt % PVP/ETN) according to the Taguchi experimental design are shown in Figure 5. When the Figure 5 was analysed, it was observed that quite smooth and uniform (bead-free) nanofibers were produced from PVP/ETN solutions. In addition average fiber diameter increases as the PVP polymer concentration increases.

10 %

12 %

14 %

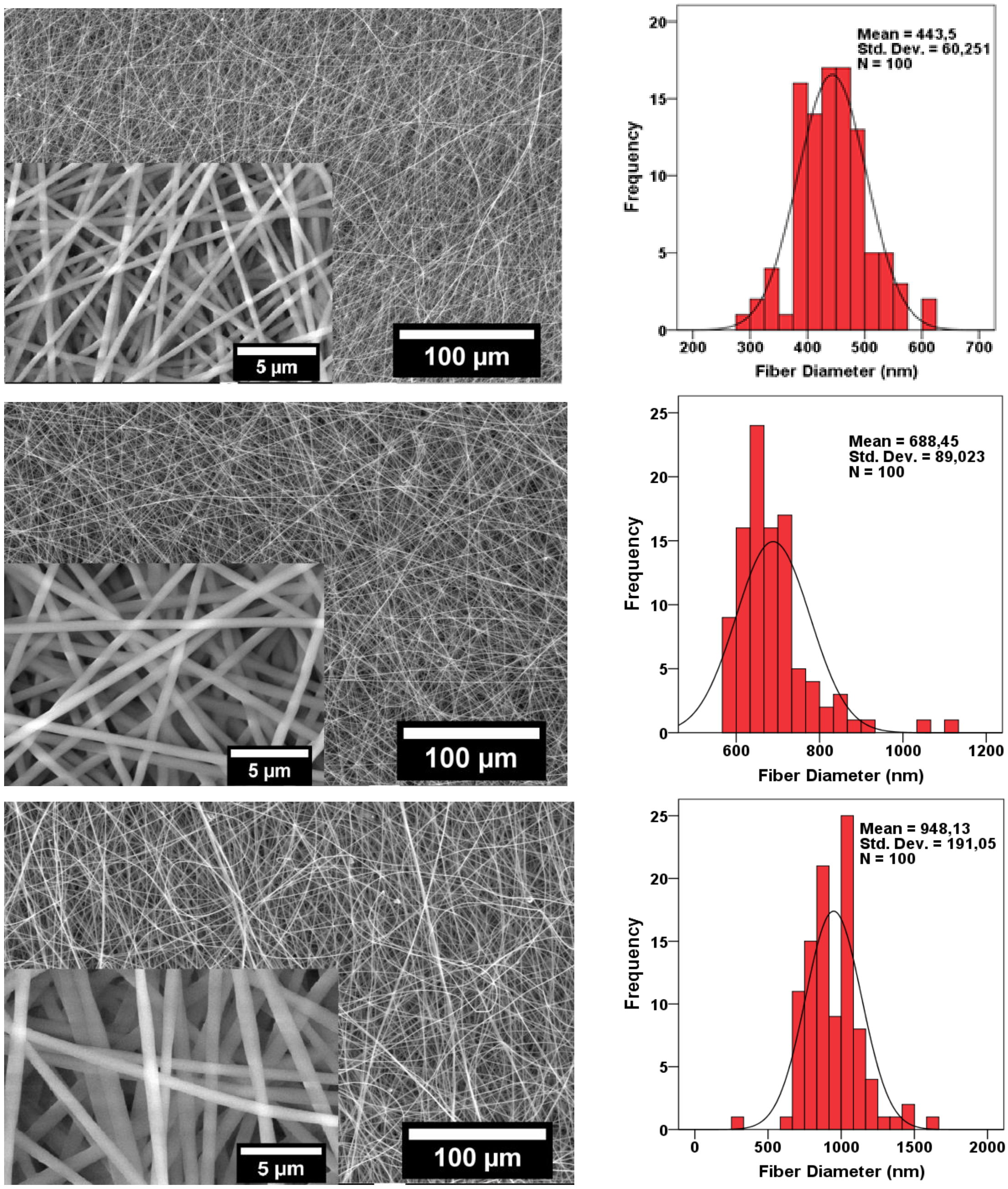

Figure 5. SEM images and histograms of nanofibers obtained from PVP/ETN solutions (1.000x and 20.000x)

Figure 6 shows that SEM images of nano fibers obtained from experiments with AA. While the nanofibers produced in the 10 % and 12 % concentrate produced by experiment 4 and 5 were observed to be quite smooth, the structure of the nanofibers formed by the PVP polymer solution prepared with 14 % AA was not homogeneous and the formation of adhesive structure was determined. Also, histogram images of these measurement results are given in the same figure.

10 %

12 %

14 %

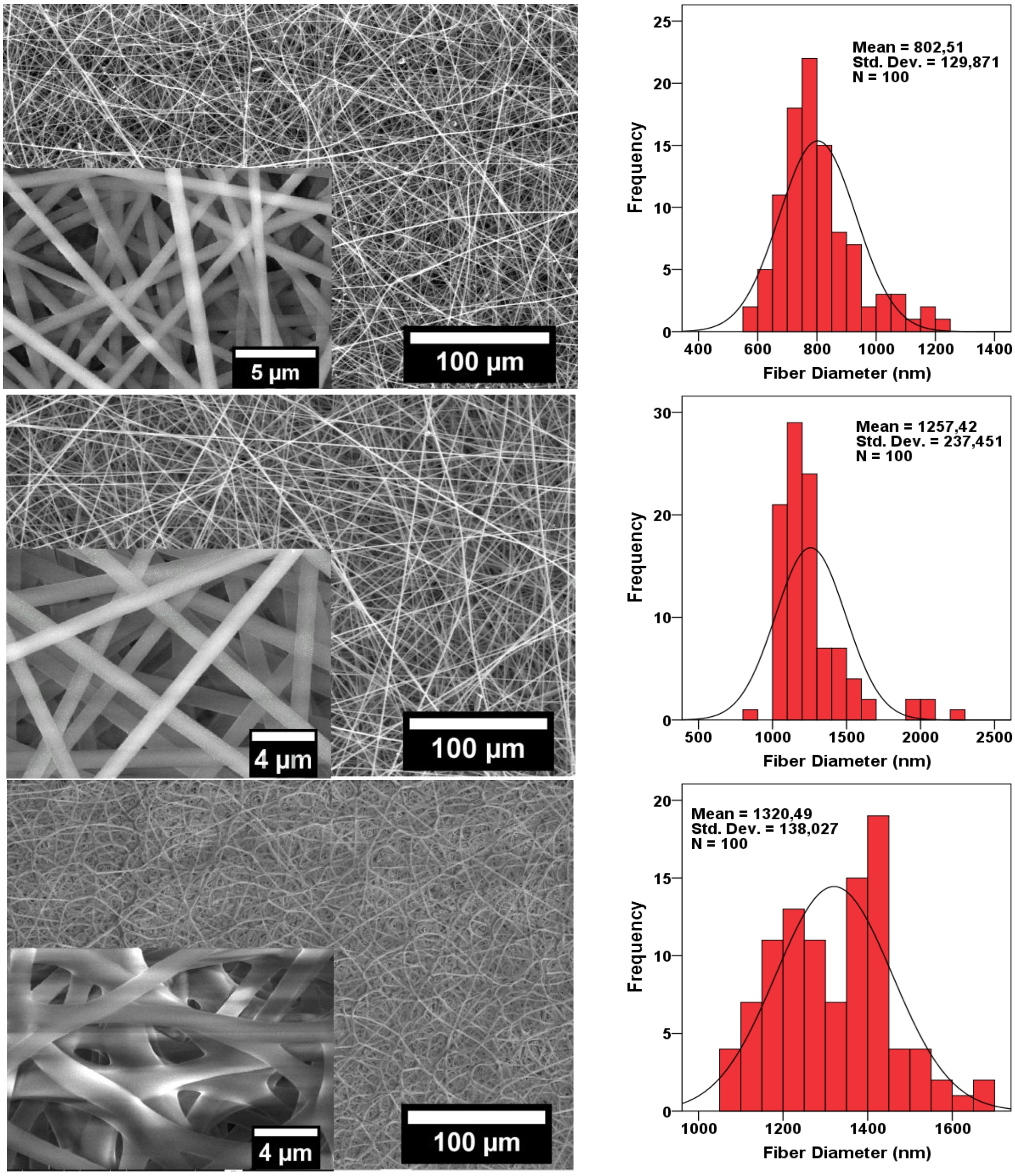

Figure 6. SEM images histograms of nanofibers obtained from PVP/AA solutions (1.000x and 20.000x)

Figure 7, shows the SEM images of the nanofibers obtained from experiments using DMF with 10 wt % PVP (experiment 7) was over-beaded, 12 wt % PVP (experiment 8) had less beaded; In experiment 9, a more uniform nano fiber structure was determined instead of beaded structure at 14% PVP. In general, the average fiber diameter of PVP/DMF nanofibers is low.

10 %

12 %

14 %

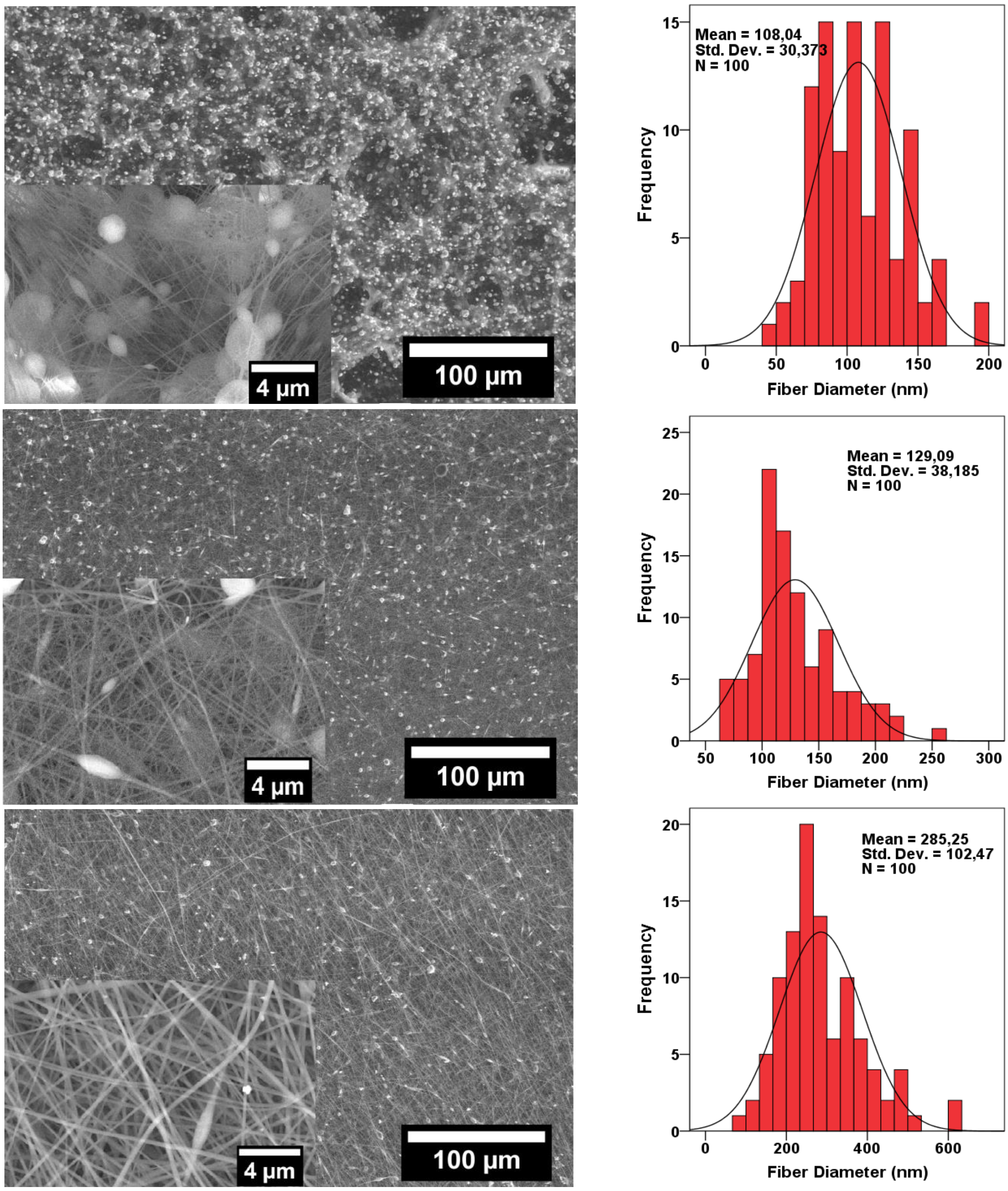

Figure 7. SEM images and histograms of nanofibers obtained from PVP/DMF solutions (1.000x and 20.000x)

Figure 8, shows the SEM images of the nanofibers obtained from experiments with CHL. It is observed that SEM images with 10% PVP, 12% PVP and 14% PVP polymer concentrations, the fiber morphology of the nanofibers are not very good in terms of fiber diameter.

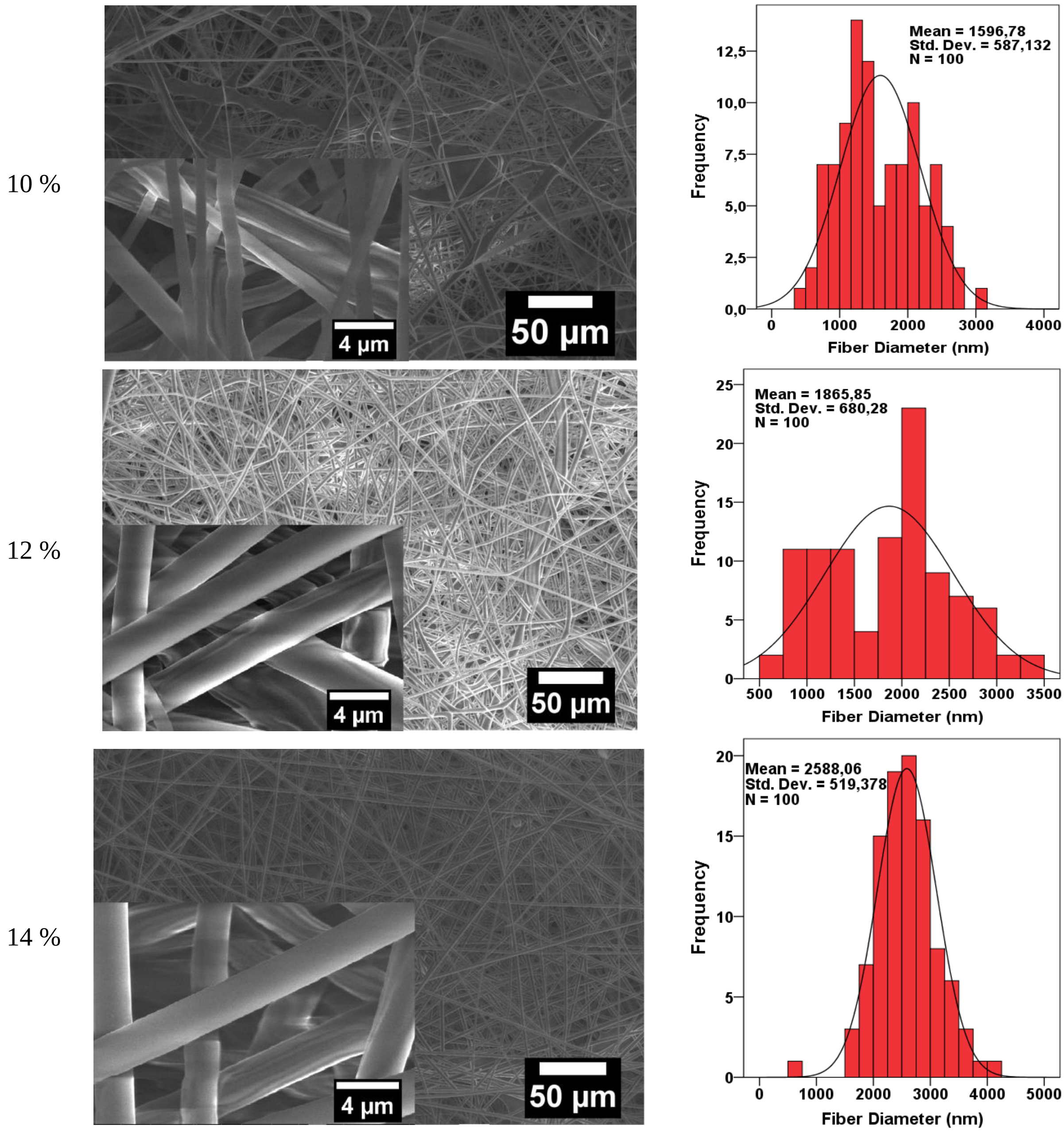

Figure 8. SEM images and histograms of nanofibers obtained from PVP/CHL solutions (1.000x and 20.000x)

Figure 9 shows the SEM images of the nano fibers obtained from experiments with DMAC. When SEM images were examined, beaded nano fiber structure was observed in 10% PVP/DMAC, 12% PVP/DMAC, 14% PVP/DMAC. Therefore it is possible to say that DMAC is not suitable solvent for PVP.

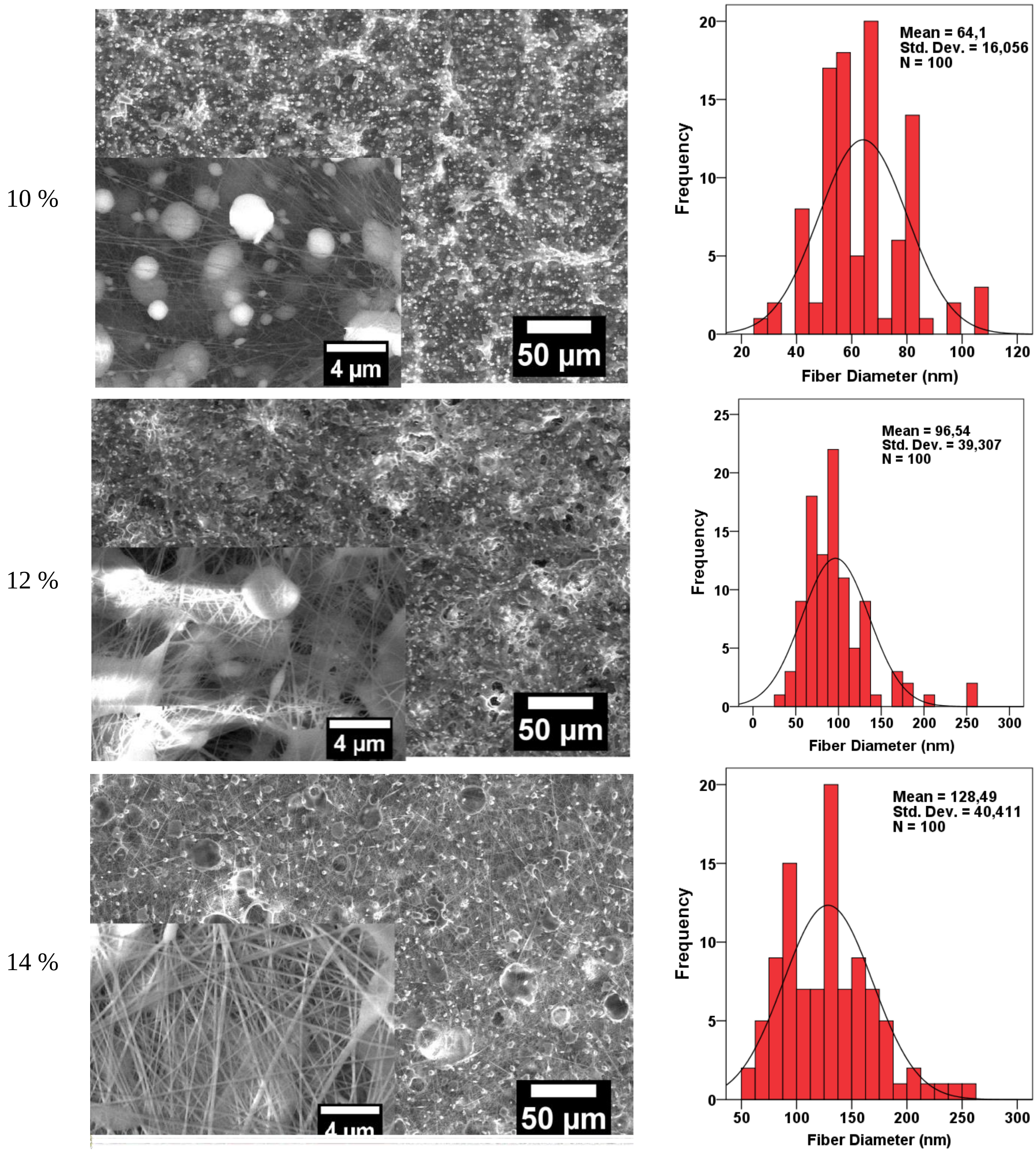

Figure 9. SEM images and histograms of nanofibers obtained from PVP/DMAC solutions (1.000x and 20.000x)

Figure 10 also shows that very intense beaded nanofiber structure of PVP nanofibers with distilled water solvent. Especially at 10 and 12 % PVP concentrations, there are very few nanofibers and very beaded structure. As the PVP concentration increases, number of bead decreases and amount of fiber increases.

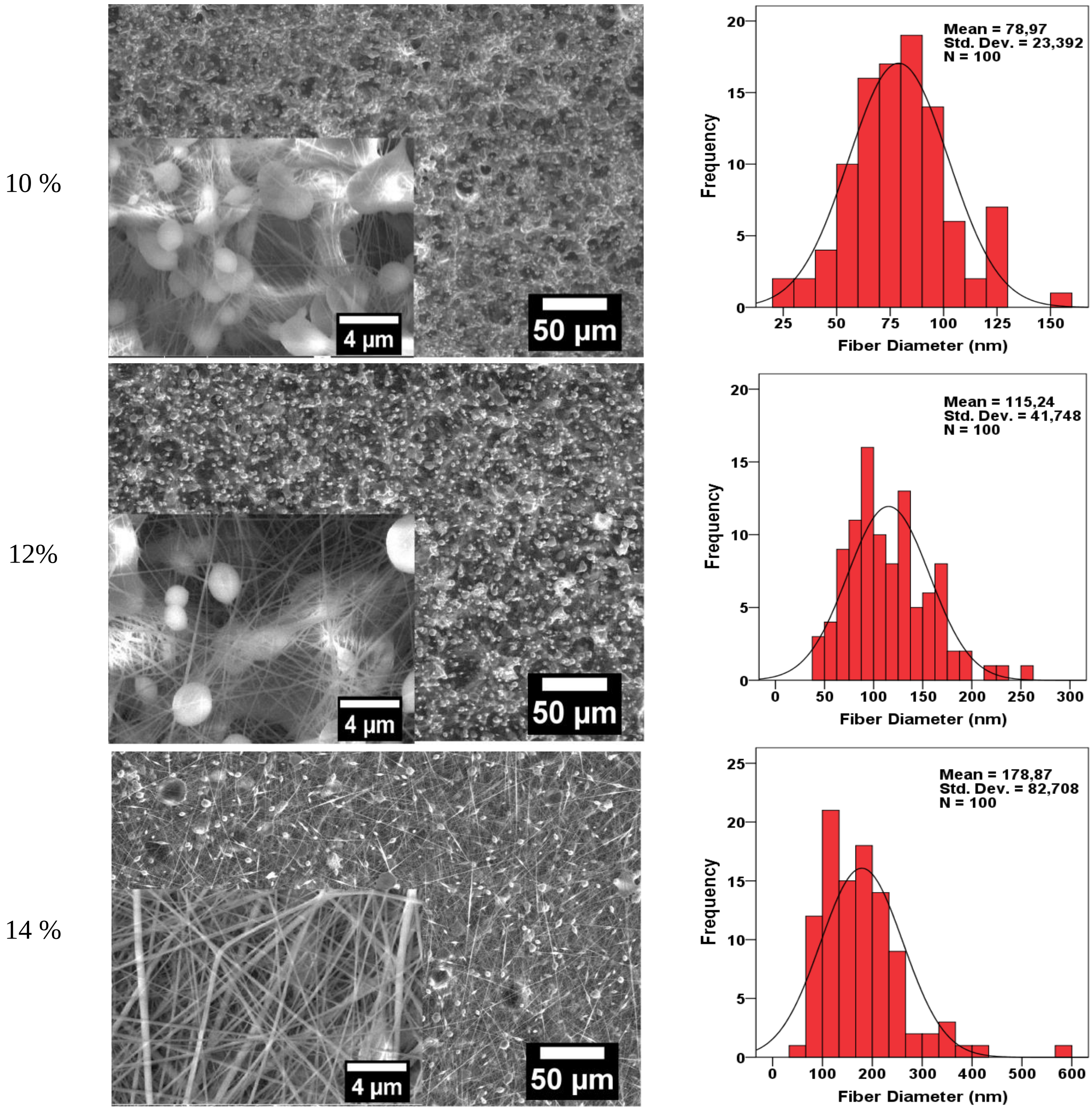

Figure 10. SEM images and histograms of nanofibers obtained from PVP/DW solutions (1.000x and 20.000x)

In the scope of the study, SEM images of fiber structures performed with 18 experiments were evaluated with the Image J image analysis program and 100 fiber samples were taken and the average fiber diameter was measured. Figure 11 shows the average fiber diameters graph obtained from 100 measurements.

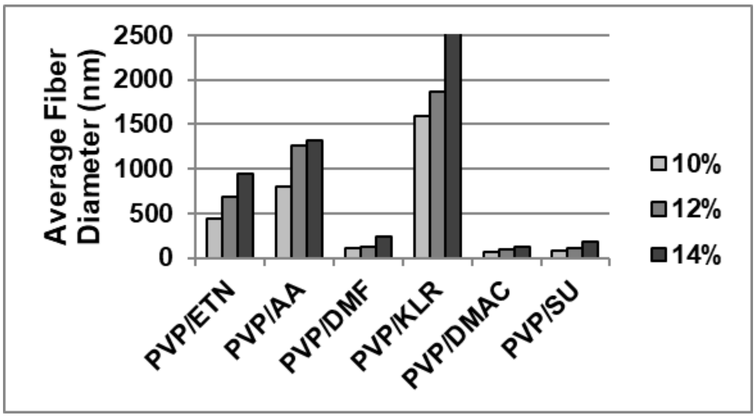

Figure 11. The results of average fiber diameter for PVP with various solvents and concentrations.

According to the Figure 11, the highest fiber diameter values were obtained with chloroform solvent. Generally microfibers were obtained with chloroform, ethanol and acetic acid solvents. On the other hand, nanofibers were obtained with DMF, DMAC and distilled water solvents. However beaded structure is a problem for DMF, DMAC and DW.

Figure 12 shows the fiber diameter uniformity coefficient measurement results for various solvents and PVP concentrations. Generally fiber diameter coefficient values are very close to 1 for all solvents of PVP. The most uniform (1,011) nanofibers were obtained from PVP/AA solution at 10 % PVP concentration.

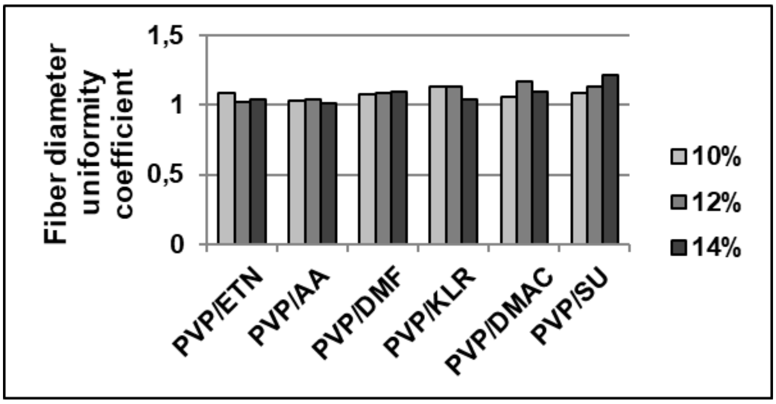

Figure 12. The results of fiber diameter uniformity coefficient for PVP with various solvents and concentrations.

According to the results obtained in Figure 13, in order to minimize fiber diameter; the most suitable values are solvent type: DMAC, polymer concentration: 10 %, applied voltage: 28.4, distance between needle tip-collector plate: 17 cm, feed rate: 0.7, humidity: 30 %.

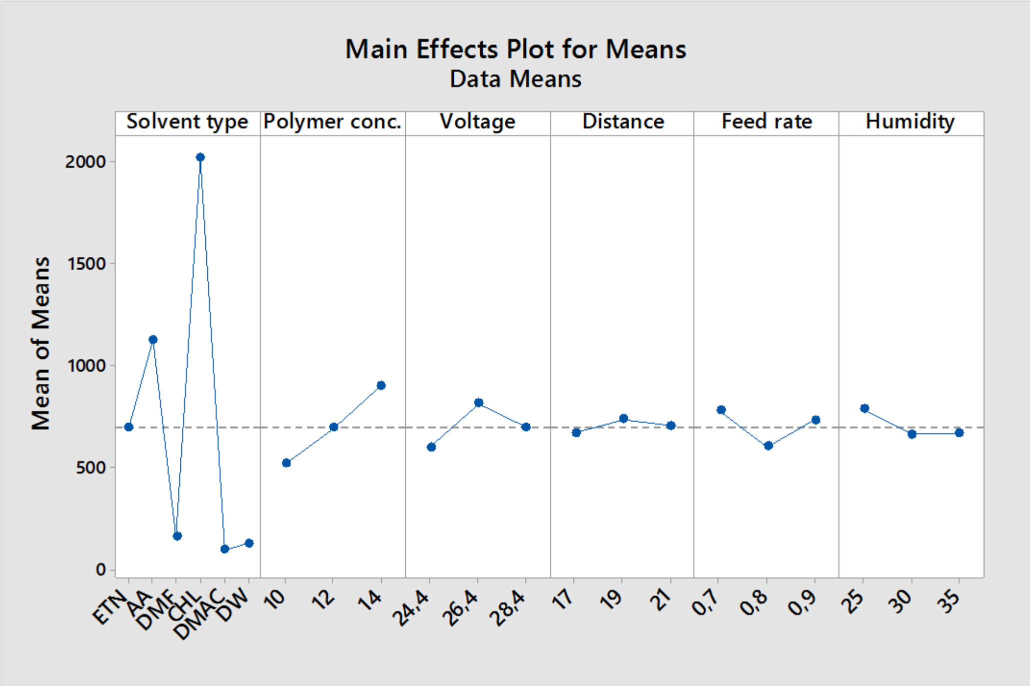

Figure 13. Effect of process parameters on S/N ratio for fiber diameter [33]

Figure 14 shows the effect of the process parameters applied on the averages for the average fiber diameter. CHL as a solvent has the highest values of 14% concentration, 26.4 kV voltage, distance between electrodes 0.7 cm, 25% humidity levels.

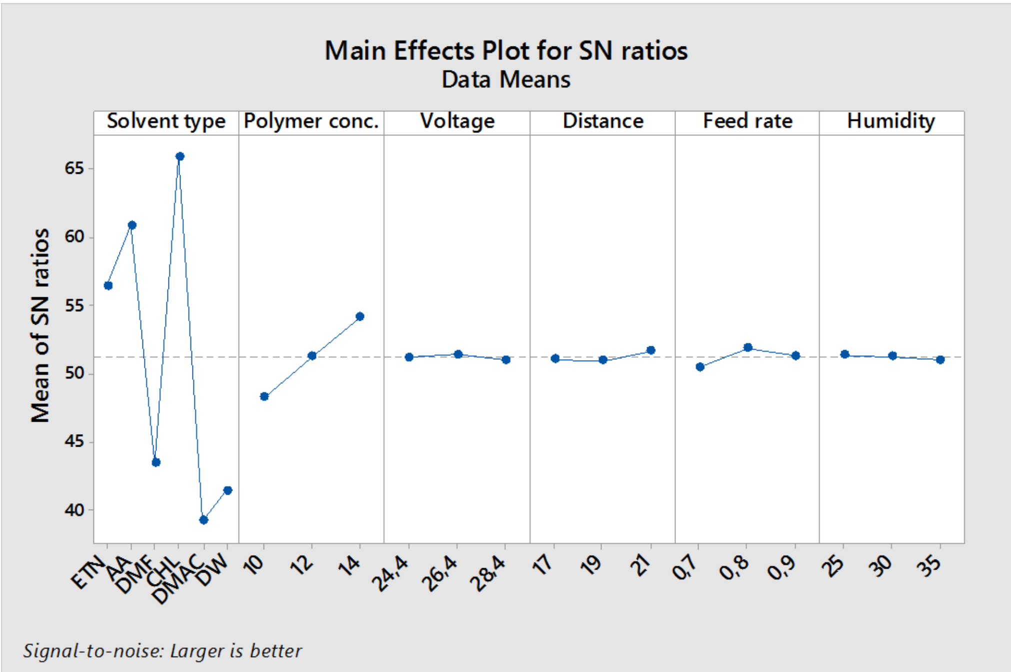

Figure 14. Effect of process parameters on averages for fiber diameter [33]

All the results of PVP solutions and fiber morphology were given in Table 3.

Table 3. All the results of PVP solutions and fibers morphology [33]

Run	Conductivity (µS/cm)	Surface tension (mN/m)	Viscosity (mPa.s)	Number average (nm)	Weight average (nm)	Fiber diameter uniformity coefficient	Fiber morphology
1	5.72	22.17	205	443.5	451.6	1.02	smooth
2	7.13	22.37	374	688.5	699.9	1.02	smooth
3	6.14	22.55	660	948.1	986.2	1.04	smooth
4	1.86	26.33	771	802.5	823.3	1.03	less beaded
5	4.66	26.26	11	1257.4	1301.8	1.04	smooth
6	2.93	16.52	2011	1320.5	1334.8	1.01	sticky
7	7.98	35.39	115	108.0	116.5	1.08	beaded
8	8.58	35.93	209	129.1	140.8	1.09	beaded
9	9.38	35.19	388	233.9	321.7	1.10	less beaded
10	0.07	24.02	96	1596.8	1810.5	1.13	sticky-smooth
11	0.07	27.55	59	1865.9	2111.4	1.13	sticky
12	0.16	-	10	2588.1	2691.3	1.04	smooth
13	6.03	34.81	143	61.1	68.1	1.06	beaded
14	6.30	34.46	287	96.5	112.4	1.16	over beaded
15	5.74	34.43	502	128.5	141.1	1.10	over beaded
16	47.80	65.74	213	79.0	85.8	1.09	over beaded
17	53.70	64.83	408	115.2	130.2	1.13	beaded
18	57.20	61.26	726	178.9	216.7	1.21	beaded

4. Conclusion

In this research, Taguchi method was applied which brings a different perspective to experimental design of electrospinning. Experiments of electrospinning were designed to minimize nanofiber diameters and at the same time to produce uniform fiber morphology. The impact of 6 parameters was examined using Taguchi's L18 Orthogonal Arrays (6 variables, 1 factor 6 level and 5 factors 3 level each). PVP micro/nanofibers with diameter ranging from 61,10 to 2588,06 µm have been found. Solvent type and the concentration were obtained as the main parameters that have a significant influence on the average diameter of the fibers. ETN as a solvent and the concentration of 12 wt % provided minimum fiber diameter. According to the results, the average fiber diameter of the fibers obtained from PVP/AA and PVP/CHL solutions can be considered as micro fibers with 1257.42 and 1320 µm respectively. The finest fiber formation is seen in fibers obtained from 10% polymer concentration of PVP/DMAC (61 µm) and PVP/WA (78 µm) with beads. At the same time, the average fiber diameters increase as the PVP concentration rises. For the fiber diameter uniformity coefficient results, the most uniform nano fibers were obtained from 10 wt % PVP/AA (1.011) and 12 wt % PVP/ETN (1,016) solutions.

As a result of the calculations, the effects of all factors on performance were observed. MINITAB 2018 program was used to find the best values of the factors and levels and it was estimated that A1B2C5D6 levels would give the best value according to the results. However, since the solvent type in the L18 orthogonal arrangement is 6 levels, it suppresses the other 5 factors in 3 levels and prevented its effects.

Author Statement

Aysun Pınarbaşı: Methodology, Investigation, Original Draft Writing, Visualization, Formal Analysis.

Funda Cengiz Çallıoğlu: Investigation, Resource/Material/Instrument Supply, Review and Editing, Supervision, Observation, Advice, Project Administration

Acknowledgment

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Conflict of Interest

As the authors of this study, we declare that we have no declarations of conflict.

Ethics Committee Approval and Informed Consent

It is not necessary to get ethics committee approval and informed consent

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