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The Effects of The Distance Between Needle And Collector Plate On The Morphology Of Fibers Produced By The Electrospinning Method

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Abstract: In the study, the morphology of the fibers produced by electrospinning using polyacrylonitrile polymer was investigated by changing the distance between needle and collector plate. For this purpose, 8 cm, 13 cm, 18 cm, 23 cm, 28 cm distances were studied. With the experimental parameters applied in the study at 8 cm and 13 cm distances, continuous fiber formation did not occur and a dense dripping was formed. When the distance was increased to 18 cm, fiber production started without interruption. The diameters of the nanofibers were measured by scanning electron microscopy (SEM), and the SPSS program was used to compare the diameter values obtained statistically. When the distance is 18 cm, the average diameter of the produced nanofibers varies between 509.96-572.48 nm, while this value varies between 460.90-522.01 at 23 cm and 399.67-462.48 at 28 cm. It was observed that the fiber diameter decreased as the distance between needle and collector plate increased. As the distance decreases, the fibers are gathered together more on the paper surface. Therefore, a thicker fiber layer was obtained when the distance was 18 cm. In addition, nanofiber fineness optimization was carried out according to the Taguchi method with two parameters using the Minitab program.

Keywords: Electrospinning, Distance between electrodes, Nanofiber diameter, Nanofiber morphology, Optimization.

1. INTRODUCTION

Electrospinning method is a method that can be applied quite easily, but there are many parameters that affect the formation and morphology of nanofibers. The parameters affecting the nanofiber production can be grouped as follows:

Solution or melt parameters; polymer structure, molecular weight, viscosity, conductivity, concentration, pH, surface tension, temperature.
Process parameters; distance between needle and collector, needle diameter, solution flow rate, voltage, collector plate type and thickness, collector type.
Environmental parameters; temperature, humidity, pressure, type of atmosphere [1-5].

The most negative aspect of the electrospinning method is that many parameters affecting the morphology of nanofibers [6]. The distance between needle and collector is one of the parameters affecting the nanofiber morphology. As the distance between needle and collector plate increases, the path of the jet increases and finer fiber formation is expected. Moreover, the time taken for the solvent to evaporate increases and the fibers are collected on the plate in a dry way. As the distance decreases, the effect of electrostatic forces increases and the jet accelerates. Therefore, the solvent cannot find sufficient evaporation time and fibers stuck to each other can be observed on the plate [7]. The distance between collector and needle is a parameter that affects the electric field strengths and flight time. An appropriate distance should be determined by considering the evaporation time of the solvent [1, 8]. In 1939, a design was made in which the distance between the needle and the collector could be adjusted. Because when the distance was short, the solvent could not evaporate completely and the fibers could stick to each other, to the plate. With the design, this situation has been eliminated [9]. When the distance is too long, the forces that will provide fiber drawing do not occur. In this case, beaded fibers can be obtained. Therefore, the optimal distance needs to be adjusted [10,11].

Various studies have been done on the effects of the distance between needle and collector on the nanofiber morphology. There are studies in which the distance is effective on fiber morphology and fiber diameter decreases as the distance increases. Kozanoğlu (2006) worked with polyvinylalcohol (PVA) and polypropylene (PP) polymers in this study. It is stated that the fiber diameter decreases with the increase in the distance between needle and collector [12]. İkiz (2009) worked with PVA polymer. It was monitored that the fiber diameter decreased when the distance between needle tip and collector increased. Depending on the increase in the distance, the residence time of the fibers and the effect time of the forces applied to the fiber increase, so the fiber diameters decrease [13]. Üstün (2011) used PVA and PAN polymers. It is stated that with the increase in the distance between feeding unit and collector, the fiber diameters decrease and they are collected in a drier state [1]. Beypazar (2013) worked with PVA and PAN polymers. It was viewed that the fiber diameter generally decreased as the distance increased [14]. Sabit (2019) worked with PAN polymer. In the study using steel and aluminum collector, the fiber diameter decreases as the distance between injector and collector increases. It is stated that finer nanofibers are obtained as the distance increases, as the time the jets travel towards the collector will increase.

Along with these studies, there are also studies where the fiber diameter increases as the distance increases [15]. Du et al. (2008) worked with PAN polymer in their studies. They stated that the diameter of the fiber increased with the decrease in the electrostatic field strength as a result of the increase in the distance between needle and collector [16]. According to Miri et al. (2016) examined the effect of the distance on diameter of the zein fibers by varying the distance between the needle and the collector between 10 and 20 cm. They observed that the diameter of the fibers did not change significantly. However, the fiber diameter increased as the distance increased. They attributed this to the decrease in the electrostatic field strength, thus less stretching of fibers [17]. Özkoç (2010) worked with Polyacrylonitrile (PAN), Polyvinyl alcohol (PVA) polymers. When the PAN polymer is used, when the distance between needle and collector is changed between 5-15 cm, there is a decrease in fiber diameter, and when it is changed between 15-25 cm, there is an increase. Small decreases in fiber diameter were observed with increasing distance between needle and collector when PVA polymer was used [18].

There are also studies showing that distance has no effect on fiber morphology and has no significant effect. Chen et al. (2009) stated in their study to model the average diameter of poly(methyl methacrylate) nanofibers that the distance between needle and collector has no effect on the fiber diameter [19]. According to Abuzade et al. (2012) produced by adjusting the distance between needle and collector plate to 12 and 15 cm. They say that there is no significant difference between the mean nanofiber diameter and nanofiber size distribution in the two cases. When the distance between needle and collector plate was low, it was expected that the nanofiber diameter would decrease due to the increase in the electric field intensity. However, the solvent did not evaporate completely. In this case, the applied voltage was high enough to eliminate the effect of distance reduction [20]. Ahmadipourroudposht et al. (2015) investigated the diameter distribution during the production of ferrofluid/PVA magnetic nanofibers. In the study, it was seen that the distance parameter did not have a significant effect [21].

In this study, fiber morphology was investigated by changing the distance parameter between needle and collector plate by using polyacrylonitrile polymer. When the studies examining the relationships between nanofiber morphology and the distance parameter are examined, contradictory results are seen. The study tries to explain the contradictions on the subject and differs from other studies in this respect. Thus, it contributes to the literature in terms of providing additional results on the effect of the distance parameter between needle and collector plate on nanofiber morphology. In addition, the study also includes optimization work on fiber fineness.

2. MATERIAL AND METHOD

2.1. Material

In the study, polymer solution was prepared by dissolving PAN polymer in dimethylformamide (DMF) solvent at room temperature. The molecular weight of the PAN polymer used is 150,000 g/mol. The viscosity of the 12% prepared solution was 891 cp and the conductivity value was 116 µS\cm. Other materials used in the study are; Plastic syringe (10 ml) to feed polymer solution into system, silicone tubing (1.5 / 4 ml) to deliver the polymer solution from the syringe to the needle, glass beaker (50 ml, 250 ml, 400 ml) to be used in the preparation and storage of polymer solution, pipette (10 ml, 25 ml), glass bottle (250 ml, 500 ml), needle tip.

2.2. Method

In the study, a syringe-fed electro-spinning device was used. Figure 1 shows the electro fiber production setup used in the experiments.

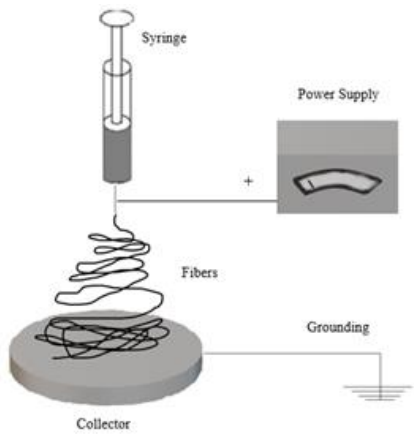


Figure 1. Syringe-Fed Electro Fiber Production

This assembly occur of three main parts: high voltage power supply, metal collector (grounded) and polymer feed pump. The voltage can be adjusted gradually with the existing power supply.

The positive end of the power supply is connected to syringe, and the negative end is connected to metal collector. An electrostatic field was created between the polymer solution drop at the needle tip and the metal collector, and the applied voltage caused polymer solution drop to be sprayed from the needle. Due to electrical forces, polymer solution drop elongated into a very thin fiber, and when the solvent evaporated, a fairly long, randomly dispersed fiber network was obtained, which accumulated on the surface. Black colored paper was placed on the collector in order to easily separate the fiber web from the surface and to examine it morphologically. The nanofibers were collected on paper for 10 minutes. All experiments were realized under normal atmospheric pressure and at room temperature. Experimental parameters are given in Tables 1 and 2.

Table 1. Experiment parameters

Process parameters	Value
Voltage Amount	25 kV
Flow Rate	1 ml/saat
Metal Collector Material	Copper
Metal Collector Thickness	10 mm
Metal Collector Shape	Circle (10 cm diameter)
Needle Diameter	22G (0.7 mm)
Distance Between Electrodes	8 cm, 13 cm, 18 cm, 23 cm, 28 cm

Table 2. Experiment parameters

Process parameters	Value
Voltage Amount	15 kV-20 kV-25 kV
Flow Rate	1 ml/saat
Metal Collector Material	Copper
Metal Collector Thickness	10 mm
Metal Collector Shape	Circle (10 cm diameter)
Needle Diameter	22G (0.7 mm)
Distance Between Electrodes	18 cm, 23 cm, 28 cm

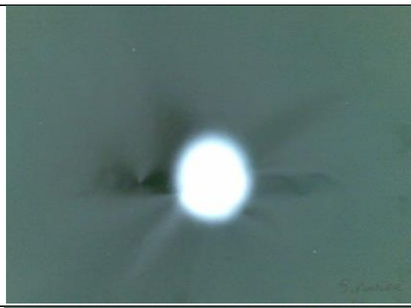
In the study, the effect of the distance between needle and collector plate on the morphology of the nano-network structure was investigated in the production of nanofibers by single-needle electro-spinning method. It was studied with the experimental parameters given in Table 1. Except for the distance between the electrodes, all parameters were kept constant and 5 different distances were studied. Samples were created by taking some amount from the middle parts of the nanofibers accumulated on the paper surface. Scanning electron microscope (SEM) in Anadolu University Materials Science and Engineering laboratory (Eskişehir) was used to determine the diameters of the produced nanofibers. The average fiber diameter was calculated by making 10 random diameter measurements for each sample and 40 diameter measurements for each different distance value. SPSS program was used to compare the fineness of the obtained nanofibers statistically. In addition, in the study, the diameter value of nanofibers was optimized according to the Taguchi orthogonal design with the Minitab program. For this purpose, the experimental parameters given in Table 2 were studied. In order to test the statistical reliability of the results obtained, analysis of variance (ANOVA) was performed with the Minitab program, using the signal/noise (S/N) ratios.

3. RESULT AND DISCUSSION

The applied voltage provides both the charge necessary for the polymer solution to be affected by the electric field and the formation of the electric field between needle and collector. Trials were started with the distance between needle and collector plate being 8 cm and 13 cm. However, it was seen that these distance values were not suitable for continuous fiber formation with the parameters given in Table 1. A dense drip occurs when the distance between needle and collector plate is 8 cm and 13 cm. When the applied voltage at these distances is set to 20 kV, dripping has decreased and fiber production has been observed. When the distance is short, the effect of electrostatic forces increases and the polymer jet accelerates. Therefore, since there is not enough time for the solvent to go away, structures that have not dried and adhered to each other can be observed on the collector [7, 15, 22, 23]. When the distance between needle and collector plate was increased to 18 cm, fiber production started uninterruptedly. When the distance is set to 28 cm, the fibers obtained spread over a wider area than the fibers produced at 23 cm and 18 cm distances (Table 3). As can be seen from Table 3, as the distance between the electrodes decreases, the fibers are gathered together more on the paper surface. Thus, when the distance is 18 cm, a thicker fiber layer is obtained compared to other distances.

As a result of SEM measurements, it was determined that there was a decrease in fiber diameters with increasing distance. When the distance is 18 cm, the average diameter of the produced nanofibers varies between 509.96-572.48 nm, while this value varies between 460.90-522.01 at 23 cm and 399.67-462.48 at 28 cm (Figure 2-4).

Table 3. The appearance of nanofibers obtained at different distances on the paper surface

Distance between electrodes	Appearance on the paper surface
8 cm	Less fiber formation, dense drip
13 cm	Less fiber formation, dense drip
18 cm	
23 cm	
28 cm	

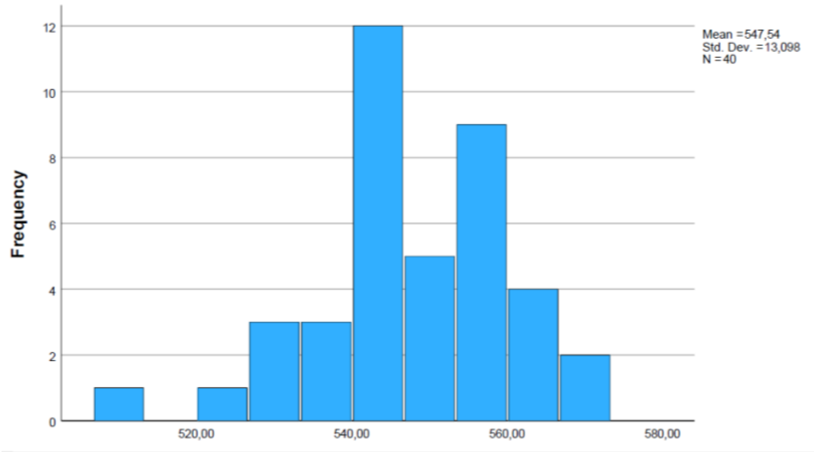


Figure 2. Diameter distribution of nanofibers obtained when there is a distance of 18 cm between needle and collector

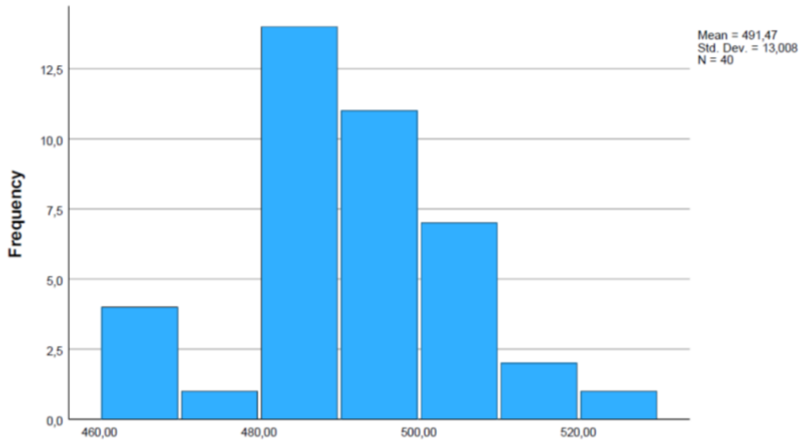


Figure 3. Diameter distribution of nanofibers obtained when there is a distance of 23 cm between needle and collector

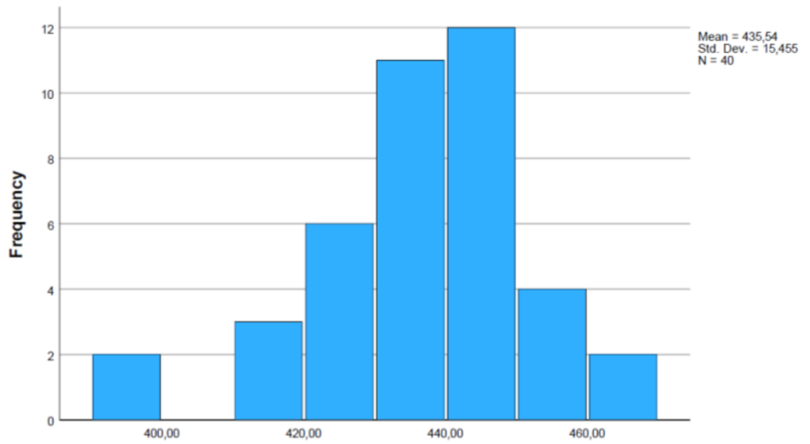


Figure 4. Diameter distribution of nanofibers obtained when there is a distance of 28 cm between needle and collector

The relationship between the average diameter values of the nanofibers obtained at different distance values can be seen in the graph created with the Microsoft Excel program (Figure 5). As seen in Figure 5, the fiber diameter decreases as the distance between the needle and the collector plate increases.

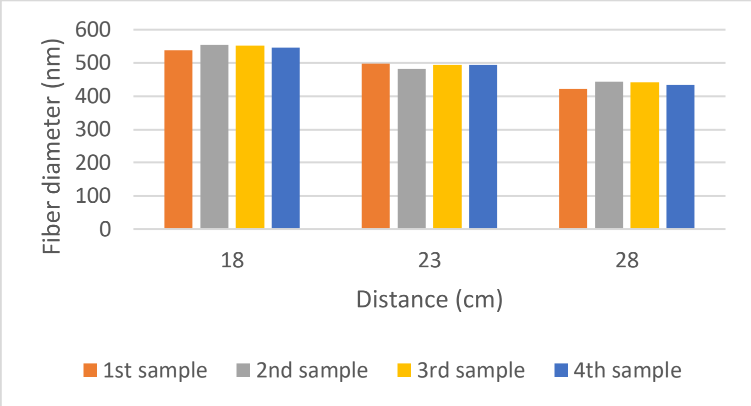


Figure 5. Average diameter values of nanofibers obtained at different distance values

Parametric tests were performed to see if the change in fiber diameter data was statistically significant. In order to apply these tests, it is necessary to meet the conditions that the data comply with the normal distribution and that the variances are homogeneous. Shapiro-Wilk test and homogeneity of variances tests were used. As seen in Table 4, diameter data have a normal distribution (p>0.05). It is seen from the same table that the variances are homogeneous (p>0.05).

Table 4. Results of Normality and Homogeneity of Variances test

	Distance (cm)	Shapiro-Wilk Statistic	df	p
Diameter	18	.971	40	.383
	23	.979	40	.667
	28	.959	40	.160

Test of homogeneity of variances

Diameter	Levene Statistic	df1	df2	p
	.725	2	117	.486

One-way analysis of variance (ANOVA) was done to determine the statistical significance of fiber diameter differences between groups for different distance values. As seen in Table 5, the differences between the levels of the distance factor were found to be statistically significant as a result of the analysis (p<0.05). Tukey test was used to determine which of the 3 levels (18 cm, 23 cm, 28 cm) showing the distance value differed in terms of the diameter averages showing the fiber fineness (Table 5). There is a significant difference in diameter between 23 cm distance and 18 cm and 28 cm distances (P<0.05). Accordingly, as the distance between needle and collector plate increases, the fiber diameter decreases statistically.

Table 5. Analysis of variance and Tukey test results

Analysis of variance					
	Sum of squares	df	Mean squared	F	p
Between groups	250880.117	2	125440.059	649.238	.000
Within groups	22605.726	117	193.211		
Total	273485.843	119			

Tukey test			
(I) Distance	(J) Distance	Average difference (I-J)	p
18 cm	23 cm	56.06625*	.000
	28 cm	112.00000*	.000
23 cm	18 cm	-56.06625*	.000
	28 cm	55.93375*	.000
28 cm	18 cm	-112.00000*	.000
	23 cm	-55.93375*	.000

The Taguchi Method is an experimental design and optimization method that includes parameter design, system design, and tolerance design. Taguchi's experimental design method is very useful for determining the optimum combination among different levels of different parameters [24]. According to Khanlou et al. (2015) show that the Taguchi method is an effective approach to statistically optimize the critical parameters of electrospinning in order to effectively tailor the resulting fiber diameters and morphology [25]. Celep and Dinçer (2017) examined the effects of various parameters on the diameters of nanofibers in their study using polyacrylonitrile polymer. To optimize these parameters, they applied Taguchi's L16 orthogonal design to the experimental design. The Taguchi method has been shown to be an effective technique for optimizing important electrospout parameters [26]. Wu et al. (2018) used the Taguchi method to design the most suitable parameters according to different quality properties for continuous electrospun yarn of polyacrylonitrile nanofiber yarn. In the study, it is stated that this method is a unique statistical method to evaluate the optimum parameters and the effects of different factors on quality characteristics [27]. On the other hand, Sorkhabi et al. (2022) investigated the effects of electrospinning parameters on diameter and morphology of polymer nanofibers according to the Taguchi DOE method. Validation experiment has shown that the mean diameter of nanofibers is close to the optimal conditions estimated by the Taguchi DOE method [28].

In the study, it was aimed to optimize the diameter value of nanofibers according to the Taguchi method. For this, 3 different distances and 3 different voltage values were studied (Table 6). Experiments were carried out with 9 trials according to the L9 orthogonal design. The signal/noise (S/N) ratio was used in the evaluation of the test results. According to the Taguchi experimental design, the average nanofiber fineness value measured for each experiment and the S/N values calculated according to the small value good are given in Table 7.

Table 6. Control factors and levels

Factor	L1	L2	L3
F1 Distance (cm)	18	23	28
F2 Voltage (kV)	15	20	25

Table 7. The results of the experiments performed according to the Taguchi orthogonal design and S/N values

Trial No	Distance	Voltage	Average nanofiber fineness (nm)	S/N ratio (dB)
1	18	15	596.56	55.5131
2	18	20	568.48	55.0943
3	18	25	547.53	54.7682
4	23	15	491.42	53.8291
5	23	20	521.32	54.3421

6	23	25	491.42	53.8291
7	28	15	482.77	53.6748
8	28	20	468.78	53.4194
9	28	25	435.53	52.7804

To see the effects of the factors and their levels on the performance characteristics, the factor effects are given with a graphical representation and the optimum combination that gives the lowest nanofiber diameter value is determined (Figure 6). When Figure 6 is examined, it is seen that the distance between needle and collector plate has a greater effect on the nanofiber fineness than the voltage. The values with the highest S/N ratios give the optimum design. In this design, the optimum point distance is 28 cm and the voltage is 25 kV.

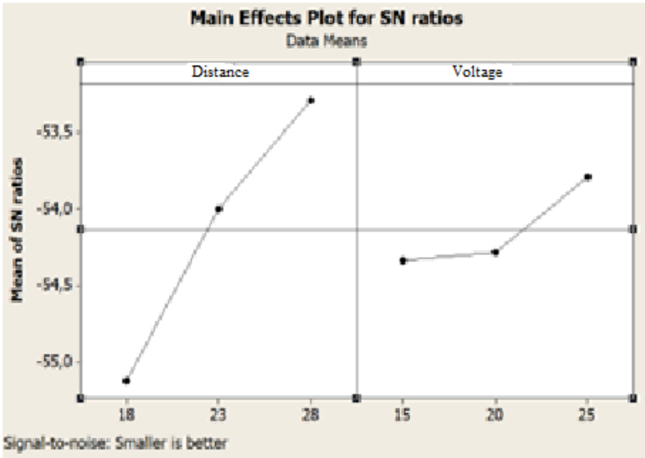


Figure 6. Graphical representation of factor effects

To test the statistical reliability of the results obtained, an analysis of variance (ANOVA) at 0.05 significance level was performed using the S/N ratios (Table 8). When the values in Table 8 are examined, the fact that the (p) value is below 0.05 indicates that the factor evaluated has a significant effect on the result value. In addition, the R^2 value of the model was 0.94. The explanatory power of the model is quite high.

Table 8. ANOVA table for Taguchi orthogonal design

Source	DF	Seq SS	Adj SS	Adj MS	F	p
Distance	2	5.1303	5.1303	2.5651	30.66	0.004
Voltage	2	0.5443	0.5443	0.2721	3.25	0.145
Error	4	0.3346	0.3346	0.0837		
Total	8	6.0091				
R^2 value of the model: 0.94						

4. CONCLUSIONS

The effect of the distance parameter on fiber morphology and fiber alignment was tried to be understood by the study performed at different distances between needle and collector plate.

When the distance between needle and collector plate was 8 cm and 13 cm, continuous fiber formation did not occur with the experimental parameters used in the study, and a dense dripping was formed. It was observed that when the applied voltage at these distances is reduced, the dripping decreases and fiber production begins. When the distance is short, the polymer jet accelerates with the effect of increasing electrostatic forces. In this case, the solvent cannot be removed and drips and bead formations may be encountered. In the study, when the distance between needle and collector plate was increased to 18 cm, fiber production started uninterruptedly.

It was observed that the fiber diameter decreased as the distance between the electrodes increased. In the electro spinning method, when the distance between needle and collector plate increases, the fibers go to the collector a longer path and their residence time increases. Therefore, the duration of action of the forces applied to the fibers also increases. Thus, the polymer jet elongates more and diameter of the fibers decreases, resulting in finer fibers. It is thought that the decrease in fiber diameter caused by increasing the distance is due to this situation. As a result of the analyzes made, it was seen that the decrease occurred was statistically significant.

When the appearance of the nanofiber networks obtained at different distance values on the paper surface was compared, it was seen that the distance between needle and collector plate affected morphological properties such as fiber arrangement and interfiber spacing. As the distance between the electrodes decreases, the fibers are gathered together more on the paper surface. Thus, when the distance is 18 cm, a thicker fiber layer is obtained compared to other distances.

Optimization of the diameter value of the nanofibers was carried out according to the Taguchi method. For this, 2 different parameters and 3 different levels of each parameter were studied. It was observed that the thinnest nanofiber diameter was obtained when the distance between needle and collector plate was 28 cm and the voltage was 25 kV.

In future studies, the effect of the distance between needle and collector plate can be examined by changing the solution parameters (such as different polymer, different concentration). In addition, an optimization study can be carried out with the taguchi method, taking into account all the parameters that affect the electrospinning.

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