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Investigation of the Effect of Needle Diameter and the Solution Flow Rate on Fiber Morphology in the Electrospinning Method

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Abstract
In the study, the morphological properties of polyacrylonitrile (PAN) fibers produced by electrospinning at different needle diameters and solution flow rates were investigated. For this purpose, 20G and 22G diameter needles were used. The fibres were produced at flow rates of 0.5 ml/hr, 1 ml/hr and 1.5 ml/hr. Scanning electron microscopy (SEM) was used to measure nanofiber diameters. Statistical analyzes were made with the help of the SPSS program. It was observed that finer fibers were obtained as the needle diameter decreased. As the solution flow rate increased, thicker fibers were obtained. In addition, it was observed that the needle diameter and flow rate affect the fiber arrangement and interfiber spacing.

Keywords
electrospinning, needle diameter, solution flow rate, nanofiber diameter, PAN.

1. Introduction

The electrospinning method is a very efficient and useful method for producing ultrafine polymeric fibers [1]. In its simplest form, this method consists of a high voltage power supply, collector, solution supply unit and needle [2]. In this process, high voltage is applied to the polymer liquid such that charges in the liquid are induced. When these charges reach a critical amount, the droplet at the tip of the nozzle forms a Taylor cone and emerges in jet form. The resulting jet moves towards the area with lower potential [3]. Nanofibers accumulate on the collector. Compared to other nanofiber production methods, electrospinning is a simple, cost-effective and widely used method [4,5,6]. Although it is an easy-to-apply method, there are various parameters that affect the formation and morphology of nanofibers. These are solution parameters (conductivity, surface tension, viscosity, etc.), process parameters (applied voltage, distance between needle tip and collector, polymer flow rate, needle diameter, etc.) and environmental parameters (temperature, humidity, etc.) [7, 8, 9]. These parameters affect the diameter and length of the nanofibers produced [10]. The biggest disadvantage of the electrospinning method is the large number of parameters affecting the morphology of the nanofibers produced [10]. The needle diameter and solution flow rate are also parameters that affect nanofiber morphology.

Rudletge et al. explained the relationship between the diameter of the fibers collected on a planar collector and the production parameters with the following equation [11],

$$d = \left[\gamma\varepsilon \frac{Q^2}{I^2} \frac{2}{\pi(2\ln X - 3)}\right]^{1/3} \quad (1)$$

where, d: fiber diameter, γ: surface tension, ε: dielectric constant, Q: flow velocity, I: current carried by the fiber, X: ratio of initial jet length to nozzle diameter. The equation shows that the fiber diameter largely depends on the flow rate, the current carried by the fiber, and the nozzle diameter [12]. In the study of He et al., it is stated that the fiber diameter increases with the increase of the needle diameter [13]. Similarly, in the study of Abunahel et al., it is shown that as the needle diameter decreases, the average nanofiber diameter decreases and finer fibers are obtained [14]. In another study which tried to produce polyacrylonitrile nanofibers with the minimum diameter and best morphology, it is shown that the diameter of nanofibers decreases with a decreasing needle diameter [15]. In a study using the same polymer with four different needle diameters, it is shown that an increase in needle diameter increases the average nanofiber diameters, and a lower nanofiber diameter variation is obtained in needles with larger diameters [16]. Akgül and Kılıç also showed that the average nanofiber diameter decreased with a decrease in the needle diameter from 1 mm to 0.5 mm [17]. Another study using PAN and PVA polymers showed that the mean fiber diameter increased with an increasing needle diameter [18]. Süslü stated that the diameter of the needle affects the fiber structure, and that the diameter of fibers and bead formation decrease with a decrease in the needle diameter [19]. In addition, Şahintürk remarked that as the needle diameter gets smaller, spraying the solution becomes more difficult, causing blockages and increasing bead formation [20]. Thompson et al. reported in their study that the needle diameter, applied voltage, needle-collector distance, relaxation time and viscosity had a significant effect on the change in fiber diameter, while the effect of other parameters was less [21]. Wu et al. indicate that the nozzle diameter affects the fiber diameter, and the degree of the effect is, from large to small, as follows; flow rate, draft rate,

nozzle number and draft temperature [22]. There are also studies showing that the needle diameter does not have a great effect on the average nanofiber diameter, and there is no relationship between them. Sencadas et al. stated that the needle diameter did not have a large effect on the mean fiber diameter, but it affected the diameter distribution [23]. Macossay et al. used three different needle sizes and showed that there was no correlation between the needle diameter and the average nanofiber diameter obtained [24]. Likewise, Albertan et al. used two different needle diameters in their study and pointed out that the needle diameter is not an effective parameter on the nanofiber diameter [25].

Looking at studies on the solution flow rate, Cramariuc et al. reported that the fiber diameter increased almost linearly with an increasing flow rate [26]. Similarly, in a study conducted to produce polyvinylacetate fiber, it is stated that the fiber diameter increased as the solution flow rate increased [27]. The solution flow rate determines the amount of solution required in electrospinning. In this method, the appropriate solution flow rate critical value is the value at which the Taylor cone is stable. When the flow rate exceeds the critical value, the fiber diameter and bead formation also increase. Megelski et al. stated that with an increasing flow rate, fiber diameters increased and more bead formation was observed [28]. It was shown that an increase in the solution feed rate raised the average diameters of nanofiber produced at four different solution feed rates using polyacrylonitrile polymer [16]. Beypazar showed that the fiber diameter decreases as the flow rate increased. She explained this situation with the increase in the amount of solution delivered to the needle tip depending on the increasing feed amount. Thus, the electrostatic forces that will affect the unit mass decrease, and in this case the jet slows down. When the jet stays longer in the electrostatic field, finer fibers are formed [18]. Roso et al. found that increasing the flow rate resulted in lower fiber diameters, which reversed after a certain point [29]. On the other hand, Sorkhabi et al. say that the effect of the solution flow rate is complex. They attribute this to the larger volume of polymer solution drawn from the needle tip with an increasing solution flow rate [30]. Mohammadi et al. observed a parabolic behavior when the flow rate was increased. That is, when the flow rate was increased up to a certain point, the diameter decreased, and when it was increased further, the diameter increased [31]. Svinterikos and Zuburtikudis state that the effect of flow velocity on the mean fiber diameter is insignificant [32]. Senthil and Anandhan in their study stated that the flow rate did not have a significant effect on fiber morphology when the poly(styrene-co-acrylonitrile) polymer was dissolved with n-butanol [33]. In their study, by dissolving the same polymer with dimethyl formamide, they found that the flow rate had a significant effect on fiber morphology, unlike their previous studies [34]. Çavdar states that the flow rate has an effect on the average nanofiber diameter, and the degree of this effect differs on the basis of the collector type [35]. In another study examining the effects of process parameters on diameter, it was stated that the polymer flow rate did not have a significant effect on fiber diameter [36]. Chen et al., in their study with poly(methyl methacrylate) polymer, determined the most important parameters affecting fiber diameter as concentration, temperature and the flow rate [37]. Working with the same polymer, Khanlou et al. show that the concentration and flow rate have a significant effect on the mean fiber diameter [38]. Fong et al. state that the morphology of fibers depends on the solution concentration, the distance between the tip and the collector, and the applied voltage, including the flow rate [39,40].

When we look at the studies examining the relationships between nanofiber fineness, needle diameter and the solution flow rate, it is seen that there are conflicting results. In this study, fiber morphology was investigated by changing the needle diameter and polymer flow rate parameters using polyacrylonitrile polymer. Thus, the study provides additional results and contributes to the literature by trying to explain the contradictions on the subject.

2. Materials and Methods

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 was 150.000 g/mol. The viscosity of the 12% solution prepared was 891 cP and the conductivity value- 116 µS\cm. Other materials used in the study were as follows; a plastic syringe (10 ml), glass beaker (50 ml, 250 ml, 400 ml), pipette (10 ml, 25 ml), and glass bottle (250 ml, 500 ml).

In this study, a single-needle electrospinning apparatus was used. **Figure 1** shows the electro fiber production setup used in the experiments. This assembly consists of three main parts: a 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 the syringe and the negative end to the 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 the polymer solution drop to be sprayed from the needle. Due to the electrical forces, the polymer solution drop elongated into a very fine 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 carried out under normal atmospheric pressure and at room temperature. Experimental parameters are given in **Table 1** and **2.**

In this study, the effects of needle diameter and the solution flow rate on the morphology of a nano-network structure were investigated. In order to examine the effect of needle diameter, two different diameter needle tips were used with the experimental parameters given in **Table 1**. In order to examine the effect of flow rate, three different flow rates were studied with the experimental

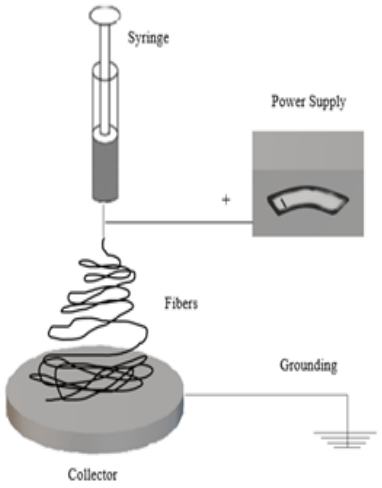


Fig. 1. Electro Fiber Production Assembly

Distance Between Electrodes	28 cm
Voltage Amount	25 kV
Flow Rate	1 ml/hr
Metal collector material	Copper
Metal collector thickness	10 mm
Metal collector shape	Circle (10 cm diameter)
Needle diameter	20G-22G

Table 1. Experimental parameters

Distance between electrodes	20 cm
Voltage amount	20 kV
Needle diameter	22G
Metal collector material	Copper
Metal collector thickness	10 mm
Metal collector shape	Circle (10 cm diameter)
Flow rate	0.5-1-1.5 ml/hr

Table 2. Experimental parameters

parameters given in **Table 2.** Samples were created by taking some amount from the middle parts of the nanofibers accumulated on the paper surface. SEM was used to determine the diameters of the nanofibers. 120 diameter measurements were made for each different parameter. The SPSS program was used to compare the fineness of the nanofibers obtained statistically.

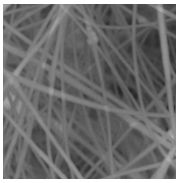
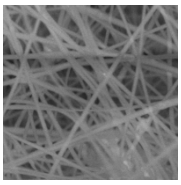
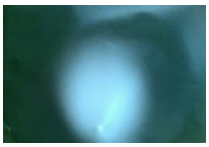
Needle	SEM images	Appearance on the paper surface
20G		
22G		

Table 3. SEM images and appearance on the paper surface

3. Results and Discussion

3.1. Effect of needle diameter on fiber morphology

When 20 gauge (0.9 mm) diameter needles were used in the study, it was observed that nanofibers spread over a wider area on the paper surface than when 22 gauge (0.7 mm) diameter needles were used (**Table 3**). When the standard deviation values in Table 6 are examined, it is seen that a lower nanofiber diameter variation is obtained in the needle with a larger diameter. Uninterrupted production was possible with both needle diameters at the parameter values of the production. It was observed that when the flow rate is increased to 2 ml/hour, it can be worked without clogging with a 20G diameter needle. When working with a 22G needle at this flow rate, there was a blockage and an increase in the amount of dripping was observed. As the needle diameter got smaller, spraying of the solution became more difficult, causing blockages and increased bead formation. When the voltage was increased at those parameters, the dripping disappeared. It was concluded that when the feed rate is increased during nanofiber production, the voltage value should be increased in order for the production to take place.

Histogram graphs showing the diameter distributions of nanofibers obtained with needles with different diameters are given in **Table 4**. As can be seen, the fiber diameters obtained when using 20 gauge diameter needles are larger than those obtained when 22 gauge diameter needles are used. As the needle diameter increases, the fiber diameters also increase.

Parametric tests were used to see whether the change in fiber diameter data was statistically significant. In order for these tests to be applied, the data must comply with the normal distribution and the variances must be homogeneous. The Kolmogorov-Smirnov test was used to determine the fit for normal distribution, since our data number was greater than 50. Test results are given in **Table 5**. It was tested whether the variances were homogeneous (**Table 5**). The values in the significance column of the table are greater than 0.05, indicating that the data have a normal distribution and their variances are homogeneous.

An independent sample t-test, one of the parametric tests, was used to determine the statistical significance of fiber diameter differences for different needle diameter values. As seen in **Table 6**, the differences between the levels of the needle diameter factor were found to be statistically significant ($P<0.05$). Accordingly, as the needle diameter increases, the increase in fiber diameter is statistically significant. It is seen that needle diameter is an effective parameter on nano fiber diameter.

3.2. Effect of solution flow rate on fiber morphology

Studies started with flow rates of 0.1 ml/hr and 0.3 ml/hr. However, it was observed

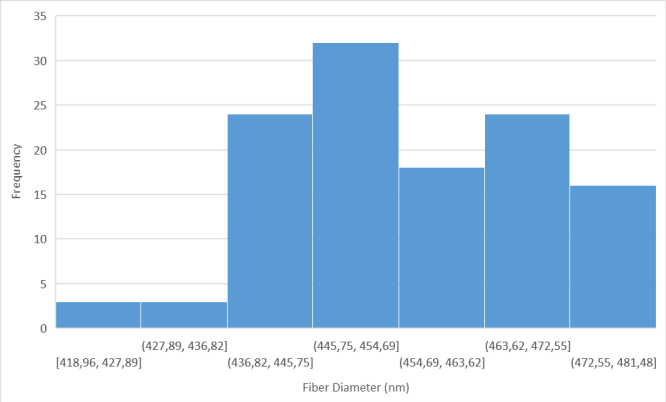
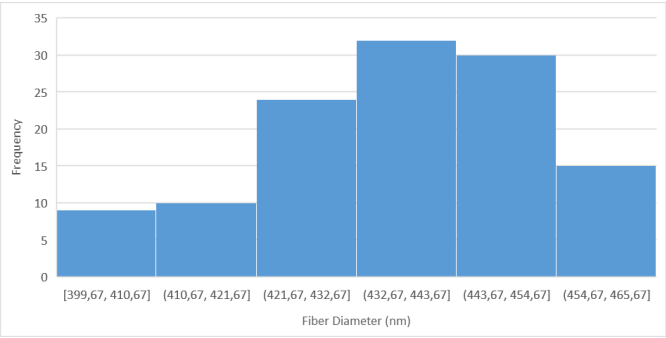
Needle	Histogram graphs
20G	
22G	

Table 4. Histogram graphs of nanofibers obtained with different diameter needles

Diameter	Needle diameter	Kolmogorov-Simirnov Statistic	df	Sig.
	20G	0.97	120	0.42
	22G	0.96	120	0.25
Test of homogeneity of variances				
Diameter		**Levene Statistic**	**df**	**Sig.**
		0.43	238	0.32

Table 5. Normality test and test of homogeneity of variances

Group	N	Mean	Standard Deviation	t	df	Sig.
20G	120	456.054	13.67	4.320	238	0.001
22G	120	435.287	15.44			

Table 6. t-test results

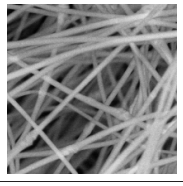
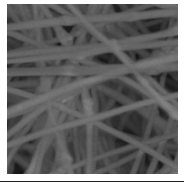
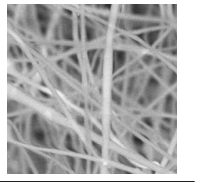
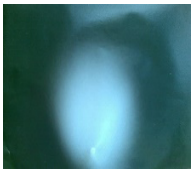
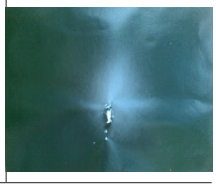
Flow rate	0.5 ml/hr	1 ml/hr	1.5 ml/hr
SEM			
Appearance on the paper surface			

Table 7. SEM images and appearance on the paper surface

that these flow rates are not suitable for continuous fiber formation with the parameters given in **Table 2.** Continuous fiber formation was achieved at a flow rate of 0.5 ml/hour. The polymer feeder was operated at flow rates of 0.5 ml/hr, 1 ml/hr, and 1.5 ml/hr. The fibers obtained at a flow rate of 1 ml/hour spread over a wider area than those produced at a flow rate of 0.5 ml/hour. When operating at a flow rate of 1.5 ml/h, dripping occurred and fiber formation was reduced (**Table 7**). It was observed that the bead formation increased when the flow rate exceeded a certain value. This is thought to be due to the fact that the solvent did not have enough time to evaporate. In addition, with an increase in the flow rate, more polymer may accumulate in the needle than necessary.

Histogram graphs showing the diameter distribution of nanofibers obtained at different flow rates are shown in **Table 8**. As can be seen, the fiber diameter increases as the flow rate increases.

Parametric tests were performed to see if the change in fiber diameter data was statistically significant. In order for these tests to be applied, the data must comply with the normal distribution and the variances must be homogeneous. The Kolmogorov-Smirnov test was used to determine the fit for normal distribution. As seen in **Table 9**, the diameter data have a normal distribution (P>0.05). It is seen from the same table that the variances are homogeneous (P>0.05).

One-Way Anova was used to determine the statistical significance of the fiber diameter differences between the groups for different flow rate values. As seen in **Table 10**, as a result of the analysis, the differences between the levels of the flow rate factor were found to be statistically significant (P<0.05). Tukey's test was used to determine which of the 3 levels (0.5 ml/h, 1 ml/h, 1.5 ml/h) showing the flow rate were different in terms of the diameter averages showing the fiber fineness (**Table 10**). There is a significant difference in diameter between flow rates of 1 ml/hr and those of 0.5 ml/hr and 1.5 ml/hr (P<0.05). Accordingly, as the flow rate increases, the fiber

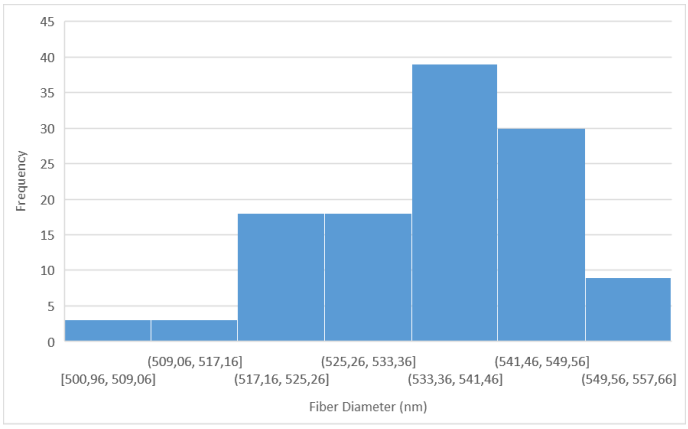
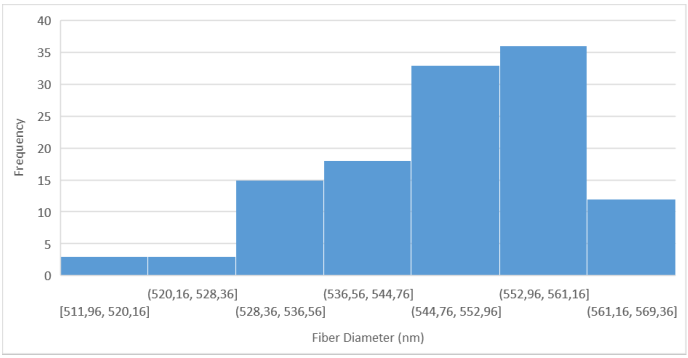
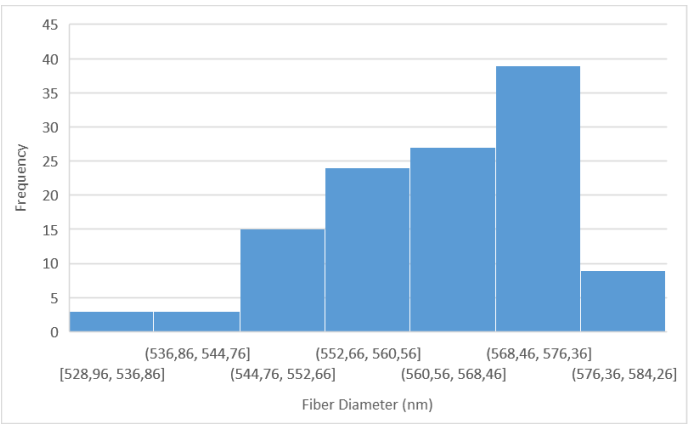
Flow rate	Histogram graphs
0.5 ml/hr	
1 ml/hr	
1.5 ml/hr	

Table 8. Histogram graps of nanofibers obtained at different flow rates

	Flow rate	Kolmogorov Simirnov statistic	df	Sig.
Diameter	0.5 ml/hr	0.96	120	0.09
	1 ml/hr	0.94	120	0.14
	1.5 ml/hr	0.94	120	0.20

Test of homogeneity of variances

	Levene statistic	df	Sig.
Diameter	0.56	358	0.89

Table 9. Normality test and test of homogeneity of variances

diameter increases statistically. The flow rate affects jet velocity and the material transfer rate. An increase in the flow rate reduces the electrostatic force acting on the transferred solution volume, and thus increases the fiber diameter [41-43]. In general, a low flow rate is recommended for the polymer solution to reach polarization in sufficient time. In addition, with an increase in the flow rate, there may be excessive polymer accumulation in the needle [44]. When the flow rate is too high, coarse granular fibers will form due to the larger volume of solution drawn from the nozzle tip. This is due to the short drying time and low tensile force before reaching the collector plate [45, 46]. If the solution flow rate is not sufficient to allow sufficient time for the solvent to evaporate, the fibers do not dry until they reach the collector and stick at the points where they come into contact with each other [47]. Therefore, the solution feed rate should be low enough for the solvent to have enough time to evaporate [3].

4. Conclusion

With the study performed at different needle diameters and solution flow rates, the effect of these parameters on the fiber diameter and fiber arrangement was attempted to be understood.

Ramarkrishra et al. stated that the inner diameter of capillary tubes such as needles and pipettes, which ensure the delivery of the solution to the drafting zone, has a significant effect, and that finer fibers can be formed with a smaller needle diameter [3]. The average fiber diameter obtained with 20 gauge needles is greater than that obtained with 22 gauge needles. As the needle diameter increases, the nanofiber diameter increases statistically. This can be explained by the enlargement of the droplet formed at the needle tip due to the enlargement of the needle diameter. As the droplet formed at the needle tip grows, the surface tension will decrease. When the surface tension decreases, the jet will accelerate more with the current applied voltage. As a result, thicker fibers will be obtained as the time the jet travels

One-Way Anova		Sum of squares	df	Mean squared	F	Sig.
	Between groups	45950.16	2	22975.08	175.93	0.000
	Within groups	46620.39	357	130.59		
	Total	92570.54	359			
	(I) Flow rate	(J) Flow rate	Average difference (I-J)	Sig.		
Tukey test	0.5 ml/hour	1 ml/hour	-11.97	0.000		
		1.5 ml/hour	-27.59	0.000		
	1 ml/hour	0.5 ml/hour	11.97	0.000		
		1.5 ml/hour	-15.62	0.000		
	1.5 ml/hour	0.5 ml/hour	27.59	0.000		
		1 ml/hour	15.62	0.000		

Table 10. Analysis of variance and Tukey test results

and stretches through the air before reaching the collector will be shortened. In addition, when the appearance of nanofiber networks obtained with needles of different diameters on the paper surface is compared, it is seen that needle diameter affects morphological properties such as the fiber arrangement and interfiber spacing.

There is a statistically significant difference in fiber fineness between 0.5 ml/hr, 1 ml/hr and 1.5 ml/hr flow rates. The fiber diameter increases as the polymer flow rate increases. As a result, the solution will reach the collector without being extended by being sufficiently attracted by the electric field. In this case, the average fiber diameter will increase. The flow rate affects the jet velocity and material transfer rate. The decrease in flow rate increases the electrostatic force acting on the transferred solution volume, and thus reduces the fiber diameter [41, 42, 43]. In the literature, there are studies in which the fiber diameter decreases when the flow rate is increased. In this case, the applied voltage is considered to overcome the increase in the amount of solution. In addition, fibers obtained while producing at a flow rate of 1 ml/hour spread over a larger area on the paper surface compared to fibers produced at a flow rate of 0.5 ml/hour. It was observed that the solution flow rate affected morphological properties such as the fiber arrangement and interfiber spacing. At a flow rate of 0.5 ml/hour, the number of drops formed at the nozzle tip per unit time is lower. In this case, since the solution is attracted by the electric field with a higher voltage, it is thought that the fibers are gathered together and thinner ones obtained. When working at a polymer flow rate of 1.5 ml/h, dripping occurred and fiber formation was reduced. When working with operating parameters determined at a flow rate of 1.5 ml/hour, the solvent did not find enough time to evaporate. As a result, since the fibers could not dry until they landed on the collector, they stuck at the points where they came into contact with each other. If the solution flow rate is not sufficient to allow sufficient time for the solvent to evaporate, the fibers will stick together [47]. It is therefore thought that the solution flow rate should be low enough for the solvent to have sufficient time to evaporate.

Since solution parameters (conductivity, surface tension, viscosity, etc.) affect the formation and morphology of nanofibers, research results can be compared by preparing solutions at different concentrations in future studies. In addition, by developing the electrofiber production mechanism, nanofiber surfaces can be obtained with different polymers in optimum conditions, and various performance properties of the surfaces obtained can be evaluated according to their usage areas.

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