Research Article

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To cite to this article: Şimşek Gündüz, G. , Göktepe, F. , Alisoy, H. & Göktepe, Ö. (2023). AN INVESTIGATION OF THE EFFECT OF COLLECTOR PLATE MATERIAL AND THICKNESS ON ELECTROSPUN FIBER FINENESS INCLUDING A THEORETICAL ANALYSIS . International Journal of Engineering and Innovative Research ,5(3), 223-236 . DOI: 10.47933/ijeir.1336360

DOI: 10.47933/ijeir.1336360

International Journal of Engineering and Innovative Research

http://dergipark.gov.tr/ijeir

An Investigation of The Effect of Collector Plate Material and Thickness on Electrospun Fiber Fineness Including A Theoretical Analysis

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(**Received:** 01.08.2023; **Accepted:** 12.09.2023)

https://doi.org/10.47933/ijeir.1336360

Abstract: The aim of this study is to examine the morphological properties of fibers produced by using metal plates of different thickness and materials as collectors in electrospinning. For this, circular shaped aluminum and copper plates of 1 mm, 5 mm, 10 mm thickness were used. The results show that much finer fibers were obtained when copper collectors are used compared to aluminum collectors. When the effect of collector thickness is analyzed, it was observed that fiber fineness increased as the collector thickness increased. In addition, it was observed that collection area of fibers on collector is much larger when aluminum collector and thinner collector is used showing that collector material and collector thickness affect fiber arrangement and interfiber spacing in electrospinning. A theoretical approach is also included in this work regarding effect of collector thickness and collector material type together with its verification in the light of experimental results.

Keywords: Electrospinning, material of collector plate, thickness of collector plate, nanofiber diameter, electrospun nanofibrous surface.

1. INTRODUCTION

Electrospinning method is a widely used nanofiber production methods due to simplicity of its set-up and its flexibility enabling to work with different polymers [1]. The nanofibrous surfaces produced by electrospinning method presents some advantages such as small pores and wide surface distribution [2], high output performance [3], and ease of medical use [4]. It has been shown that surfaces obtained with nanofibers, which are produced in an easy, flexible, and low-cost way by electro spinning method, have very good performance in many areas [5]. Electrospinning is considered the simplest and cheapest method to obtain nanofibers [6]. However, although it is easy to apply, it is a complex method in theory [7].

The properties of the collector are one of the parameters that affect the nanofiber production in the electro spinning method. There are studies using different types of collectors such as flat plate [8-11], rotating disc [12, 13], rotating roller [14-16], conveyor belt [17, 18]. In addition, instead of a single collector plate, there are systems in which two collectors are used side by side and the distance between them can be adjusted. In these systems, by moving the collector in the opposite direction, the longitudinal elongation of the fibers on it is ensured, and it has been shown that the fiber diameter decreases with this longitudinal elongation [19]. Collector systems are divided into two types: stationary type and rotating type. As the relative directions of nanofibers deposited on collector affect the properties of the obtained surface, the fibers accumulated on fixed collector plate are randomly oriented. This situation also limits usage area of the obtained surface. For this reason, it has been tried to arrange the directions of nanofibers relative to each other by using different collector types, such as rotating drums and conveyor belts as mobile type collectors, parallel rings, and frames as fixed type collectors. In addition, the rotational speed of the mobile type collectors was found to be effective on the orientation of nanofibers [7]. In addition, it has been shown that when parallel copper wires containing gaps are used, the fibers are densely gathered perpendicular to the wires and the thinnest fibers are obtained compared to other collector types [20]. In another study conducted with two types of collectors as disc and plate rotating at two different speeds, it was stated that the fibers produced by the disk-shaped collector with a rotation speed of 50 rpm are more hydrophilic and have larger pores than the plate collector [21]. In a study using roller collector and plate collector, it was stated that with the increase in concentration, the fiber fineness distribution was higher in the plate collector [22]. As a result, different collector systems have been designed mainly to obtain parallel and oriented fibers and thus increasing strength, faster production in a larger area, and simultaneous production of different structures [23-25].

The material of collector plate is also an important parameter in electrospinning as generally conductive plates are used to provide electric field and these metal plates are electrically grounded. As a result, a stable potential difference occurs between the nozzle and collector plate [26]. The effect of collector material is studied by using teflon, plastic and aluminum collector, the same result could not be obtained in teflon and plastic collector [27]. In another study, aluminum and wood collector is used and a better fiber alignment is obtained with aluminum collector as would be expected [24]. Other than metal collectors, items like methanol, water, paper were also used as collectors [25]. In a study using a water tank as collector, shrinkage and roughness were observed in the fibers. When salt was added to water to increase conductivity, the roughness changed, but the shrinkage did not. Swelling was observed in the fibers when methanol storage was used as the collector [28]. The effect of different collector materials was also analyzed in general during cellulose acetate fiber production in electrospinning. When paper is used as a collector, it has been observed that fibers have a smoother surface, they repel each other electrically because they cannot fully transmit electricity to the collector and more porous surfaces can be obtained showing that fibers collected on non-conductive material generally have a lower packing density than those collected on a conductive surface due to the repulsive forces of the charges. [29] For a conductive collector, it can dissipate electric charges and reduce the repulsion among fibers, thus favoring a tightly packed and thick membrane structure while the accumulation of charges can result information of 3-dimensional fiber structures due to the repulsive forces of similar charges when a non-conductive collector is used [2]. When nanofibers reach to the grounded collector, their current electrical charge is reset. The electrical charges in nanofibers in the upper layer are reset more slowly and this affects the fiber morphology. Therefore, the electrical conductivity value of the material from which the metal collector is produced is very important [30]. In addition to the material from which the collector is produced, its geometry also affects the nanofiber morphology [31]. The effect of collector thickness and material was also studied by using collectors in square form at different thicknesses and materials in general [32]. More recently, not in nanofibrous production but nanofiber production, four different types of conical aluminum collector was used for nanofiber yarn production in electrospinning showing that fibers in electrospun yarn becomes finer as collector thickness increases [33].

In this study, flat metal collectors in circular form were used so that mainly change in fiber diameter can be investigated by using different material and different thickness of metal collector. Also, a theoretical approach is included together with verification by using experimental results. As a result, this study aims to contribute to the findings in electrospinning field which has been widely studied but having limited works in terms of effect of collector material and collector thickness so that a suitable choice of collector in electrospinning could be carried out. In other words, this study aims to contribute to the existing findings in the field of electrospinning, to facilitate the selection of an appropriate collector for electrospinning. In the study, after giving information about the material and method, firstly the experimental results were given, and then the results were analyzed together with the theoretical approach.

2. MATERIAL AND METHOD

In this study, a polymer solution was prepared by dissolving polyacrylonitrile (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 10% (wt) prepared solution was 810 cp while electrical conductivity value was 118 µS/cm.

A laboratory type single-needle electro-spinning device was used. In Figure 1, the schematic representation of electro-spinning apparatus used in experiments is given.

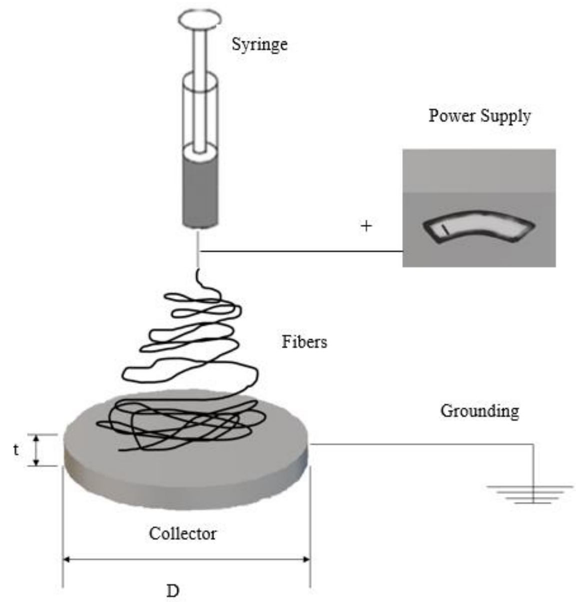


Figure 1. The schematic view of electrospinning setup used in this work.

During production, black colored paper was placed on the collector to separate fibrous web from the surface easily and clear visualization of collected area of fibers on collector surface. All experiments were carried out under normal atmospheric pressure and at room temperature. Experimental parameters are given in Table 1.

Table1. Main process parameters of electrospinning

Process parameters	Value
Distance Between Electrodes	20 cm
Voltage Quantity	18 kV
Flow Rate	1 ml/hour
Needle Diameter	22 G

As metal collectors, collector electrodes in circular form with a diameter of 10 cm but in different thicknesses and different materials were used as shown by Table 2.

Table 2. Metal collectors used in this work

Metal Collector Material	Collector Thickness (t) (mm)
Aluminum (Circular; D: 10 cm)	1
	5
	10
Copper (Circular; D: 10 cm)	1
	5
	10

In electrospinning process, four different nanofibrous surfaces were produced for each metal collectors shown at Table 2 and each nanofibrous surface was collected on the collector for 10 minutes. Then samples were taken from middle part of each nanofibrous surface for analysis. The average fiber diameter was calculated by making 10 random diameter measurements for each sample therefore resulting in total 40 measurements for each different collector thickness and collector material. To determine fiber diameters of produced nanofibrous surfaces, scanning electron microscopy (SEM) was used. For statistical analysis of experimental results, statistical parametric tests were applied to see whether the change in fiber diameters were statistically significant by using SPSS program. Shapiro-Wilk test and homogeneity of variances tests were used to determine the relevance for normal distribution and then One-Way ANOVA was used to determine the statistical significance of fiber diameter differences between groups for different collector plate thickness values and material type.

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1. Effect of aluminum collector thickness

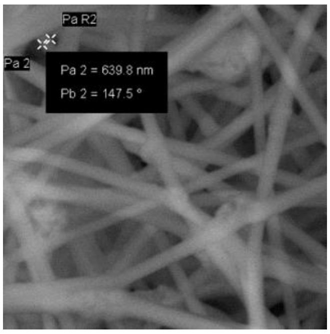
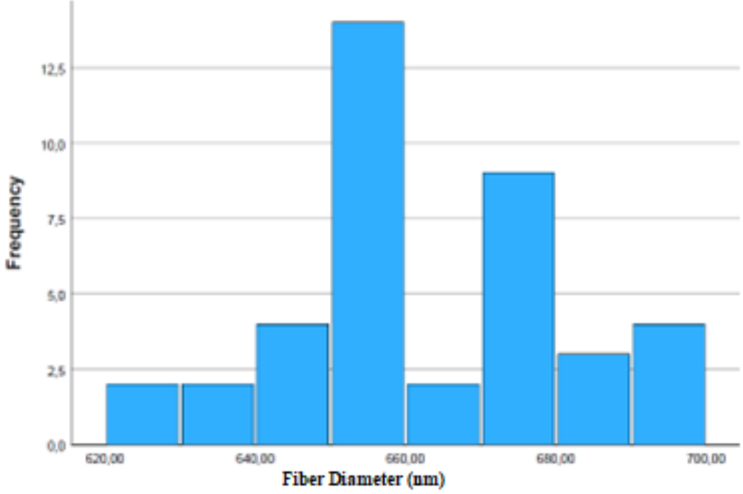
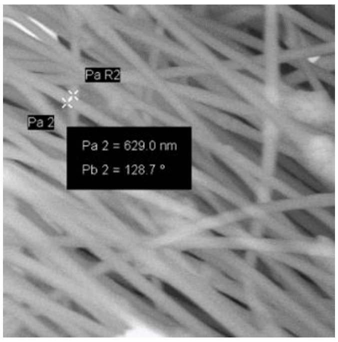
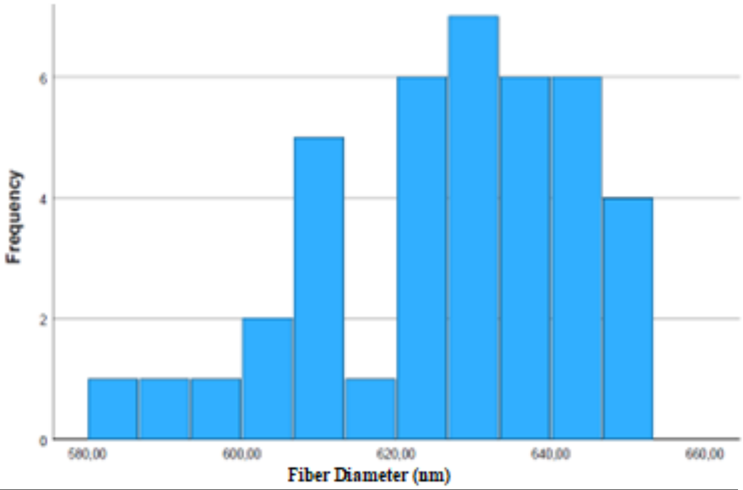
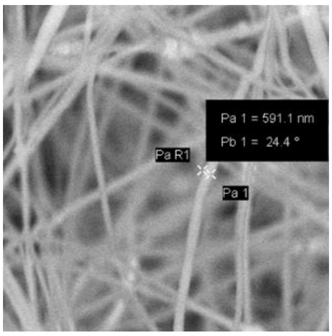
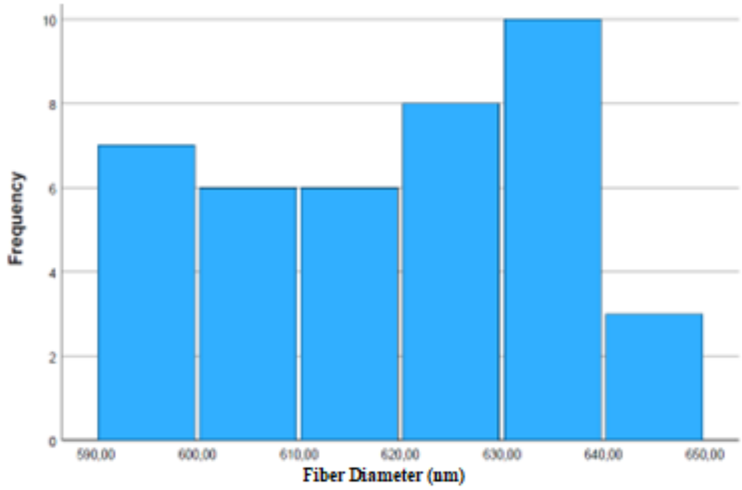
Following nanofibrous surface production, first fiber collection area on Al collector is analyzed visually in this work. It has been observed that collection is of fibers are much smaller when aluminum plate of 10 mm thickness is used compared to 5 mm and 1 mm collector plates and Figure 2 shows this situation visually.

Aluminum collector (t: 1mm) Aluminum collector (t: 5 mm) Aluminum collector (t: 10 mm)

Figure 2. Collection area of the nanofibers obtained with circular aluminum collectors of different thicknesses.

Regarding fiber fineness obtained by using aluminum collectors at different thicknesses, the typical SEM images of nanofibers produced and histogram graphics showing fiber diameter distribution are given in Table 3.

Table 3. SEM images and histogram graphs of nanofibers obtained from aluminum collector plates of different thicknesses

Al	SEM images (7,50 kx)	Histogram charts
t:1mm		
t:5mm		
t:10mm		

When statistical parametric tests were used to see whether the change in fiber diameter data statistically significant, Shapiro-Wilk test and homogeneity of variance tests was showed that fiber diameter data had a normal distribution, and their variances were homogeneous. On the other hand, the statistical analysis results are given at Table 4 as the differences between the levels of the metal collector thickness factor were found to be statistically significant ($p<0.05$). The Anova results show that there is no significant difference in average fiber diameter values for collector thickness of 10 mm and 5 mm while there is a significant difference in average fiber diameter values between collector thickness of 5 mm and 1 mm ($p<0.05$). Moreover, it can be said that fibers get finer when aluminum collector thickness increases from 1 mm to 5 mm. The geometry of the collector is also important in terms of nanofiber morphology [31]. Göktepe et al. state that the collector thickness and material difference affect the fiber diameters obtained [32]. It has been shown that the fibers obtained become thinner as the collector thickness increases [33].

Table 4. Analysis of variance and Tukey test results

Analysis of variance					
	Sum of squares	**df**	**Square mean**	**F**	**Sig.**
Between groups	41965.203	2	20982.601	68.495	.001
In groups	35841.628	117	306.339		
Total	77806.831	119			

Tukey test			
(I)Thickness	**(J) Thickness**	**Average difference (I-J)**	**Sig.**
10 Al	5Al	-8.41000	.085
	1Al	-43.20050*	<.001
5 Al	10Al	8.41000	.085
	1Al	-34.79050*	<.001
1 Al	10Al	43.20050*	<.001
	5Al	34.79050*	<.001

3.2. Effect of copper collector thickness

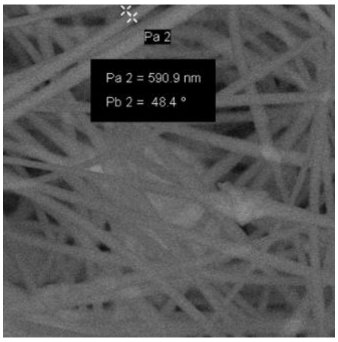
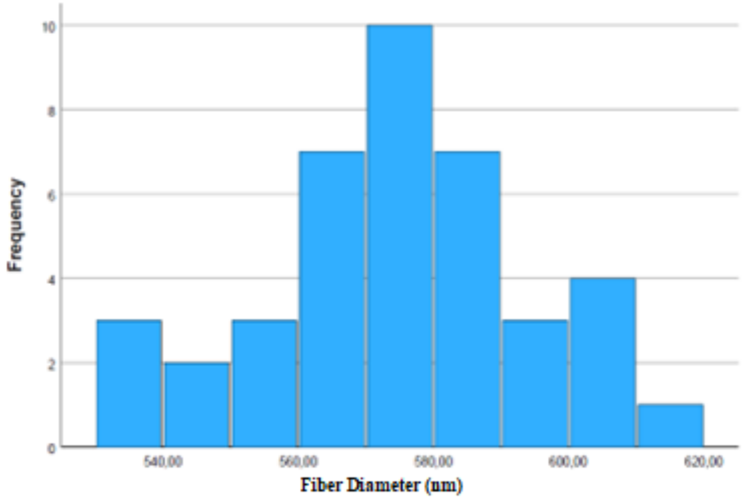
When fiber collection area on the copper collector surface is analyzed, it has been observed that fibers are collected at much smaller area when copper plate of 10 mm thickness is used as a collector compared to the collector thickness of 5 mm and 1 mm plates as this situation has been visually shown at Figure 3.

Copper collector (t: 1 mm) Copper collector (t: 5mm) Copper collector (t: 10mm)

Figure 3. Collection area of the nanofibers obtained with circular copper collectors of different thicknesses.

Following nanofibrous surface production at each collector of different thickness, fiber fineness values are analyzed. The typical SEM images of nanofibers produced using copper collectors of different thicknesses and related fiber diameter distribution histograms are given in Table 5.

Table 5 SEM images and histogram graphs of nanofibers obtained from copper collector plates of different thicknesses

<table>
<tr><th>Cu</th><th>SEM images (7,50 kx)</th><th>Histogram charts</th></tr>
<tr><td>t:1mm</td><td>
</td><td>
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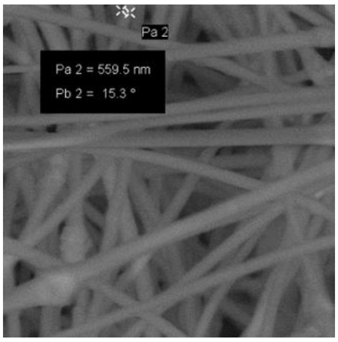
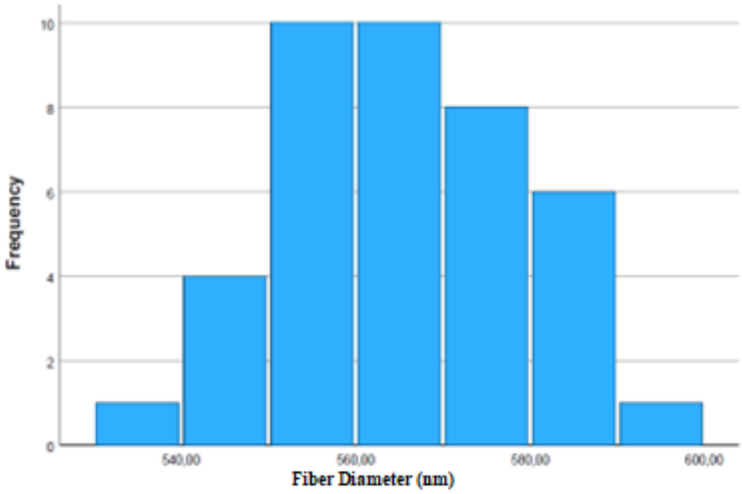
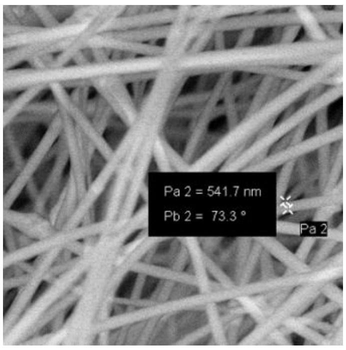
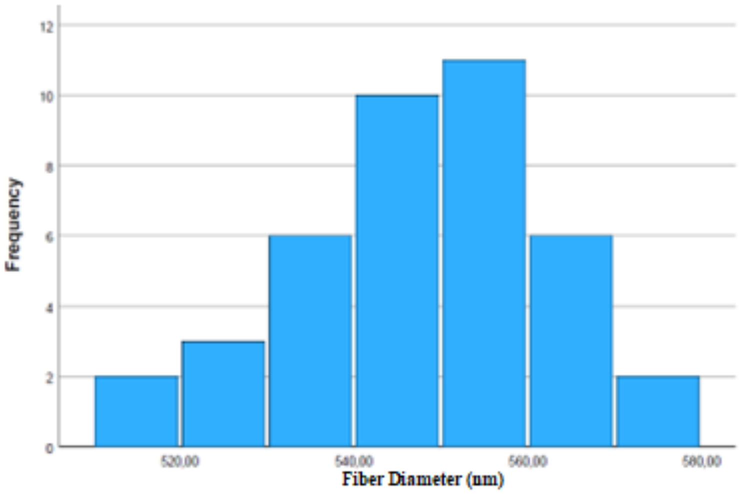
<tr><td>t:5mm</td><td>
</td><td>
</td></tr>
<tr><td>t:10mm</td><td>
</td><td>
</td></tr>
</table>

When statistical parametric tests were used to see whether the change in fiber diameter data statistically significant, Shapiro-Wilk test and homogeneity of variance tests was showed that fiber diameter data had a normal distribution, and their variances were homogeneous. One-way analysis of variance was performed to determine the statistical significance of fiber diameter differences between groups for different collector plate thickness values. As seen in Table 6, the differences between the levels of the metal collector thickness factor were found to be statistically significant ($p<0.05$). Tukey test was used to determine which of the 3 levels (10 mm, 5 mm, 1 mm), there is a significant difference in average fiber diameter between collector thickness of 10 mm and 5 mm ($p<0.05$) while there is no significant difference in fiber diameter when collector thickness of 5 mm and 1 mm is used. Moreover, it can be said that fiber diameter decreases significantly by increasing thickness of copper collector from 5 mm to 10 mm. It has been observed that as the thickness of the copper collector increases, that is, its conductivity increases, like the aluminum collector, the fibers become thinner. It is thought that finer nanofibers are obtained with much higher elongation of the jets because of the electrical conductivity increasing with the increase of the metal collector thickness.

Table 6. Analysis of variance and Tukey test results

Analysis of variance					
	Sum of squares	**df**	**Square mean**	**F**	**Sig.**
Between groups	13651.547	2	6825.773	24.764	.001
In groups	32248.587	117	275.629		
Total	45900.134	119			

Tukey test			
(I)Thickness	**(J) Thickness**	**Average difference (I-J)**	**Sig.**
10Cu	5Cu	-17.39500*	<.001
	1Cu	-25.57925*	<.001
5Cu	10Cu	17.39500*	<.001
	1Cu	-8.18425	.075
1Cu	10Cu	25.57925*	<.001
	5Cu	8.18425	.075

3.3. Effect of collector material type

The relationship between the collectors with different materials and the average diameter values of the nanofibers obtained can be seen in Figure 4 as the average diameter of the fibers obtained from aluminum collectors is higher than that obtained from copper collectors for all collector thickness values. On the other hand, the results at Figure 4 also shows that finer fibers were obtained as the collector thickness increased for both aluminum and copper collectors.

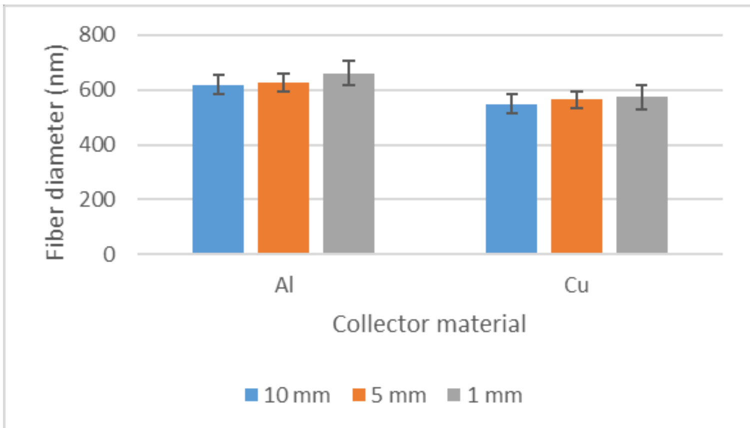


Figure 4. Average diameter values of nanofibers obtained with circular aluminum and copper collectors.

When the effect of collector material has also been analysed statistically, the independent sample t-test using average fiber diameter values obtained from 10 mm thick aluminum and copper collectors, 5 mm thick aluminum and copper collectors, 1 mm thick aluminum and copper collectors, show that differences between the levels of the metal collector material factor were statistically significant ($p < 0.05$) (Table 7). Sabit indicates that the difference in the collector material has a direct effect on the nanofiber yarn and fiber diameter [34]. The electrical conductivity value of the material from which the metal collector is produced is very important [30]. For a conductive collector, the charges on the fibers are distributed so that more fibers can be pulled into the collector and collected close together [29]. In the study, when the appearance of nanofibers on the paper surface is examined, it is seen that the fibers gather more together as the conductivity value of the collector increases.

Table 7. t-test results for comparison of average fiber diameter values obtained by aluminum and copper collector plates

Group	N	Average	Standard deviation	t	df	Sig.
10 mm Al collector	40	618.50	16.49	19.57	78	.001
10 mm Cu collector	40	548.53	15.45			
5 mm Al collector	40	626.91	16.75	17.92	78	.001
5 mm Cu collector	40	565.93	13.49			
1 mm Al collector	40	661.70	19.13	19.93	78	.001
1 mm Cu collector	40	574.11	20.14			

4. THEORETICAL APPROACH AND VERIFICATION WITH EXPERIMENTAL RESULTS

4.1. Theoretical analysis regarding fiber radius when different collector thickness is used

Regarding nanofiber production by electrospinning shown by Figure 1, the force acting unit volume of a material in such an electrical field can be stated as below [35]:

$$F = qE - \frac{1}{8\pi} E^2 \nabla \varepsilon + \frac{1}{8\pi} \nabla \left(E^2 \frac{d\varepsilon}{d\rho} \rho \right) \tag{1}$$

In Eq. (1), the first component (qE) shows the force acting on real electrical power of the material; second component is related to change in electrical permittivity of the material, ε, and the third component describes the electrostructive phenoman due to change in material density, ρ.

As can be seen by Figure 1, a high voltage is applied to the polymer solution ejected from nozzle, therefore charging polymer solution. In this case, the charge of particles in polymer jet under F force in Eq. (1) can be described as [36, 37]:

$$q_p = \left(1 + 2\frac{\varepsilon - 1}{\varepsilon + 2}\right) E_0 r_p^2 \frac{\pi nekt}{1 + \pi nekt} \tag{2}$$

where ε dielectric permittivity, E electrical field intensity, r_p radius of particle, t charging time of particles in polymer jet, n.e electrical charge density of particles and k describing mobility of charges in electrical field. In steady-state ($t \to \infty$), the limit of charge of particles in polymer jet can be shown as below where q_{max} describes the maximum charges of particles in a polymer jet:

$$q_{max} = \lim_{t \to \infty} q_p = \left(1 + 2\frac{\varepsilon - 1}{\varepsilon + 2}\right) E_0 r_p^2 \tag{3}$$

In an electrical field, the charges injected onto a solution activates surrounding polymer solution by accelarating and result as a thin polymer jet drawn towards the collector. In this case, radius of polymer jet can be described by an ampirical relation below [38, 39]:

$$r_f = \left(\frac{\rho Q^3}{2\pi^2 UI}\right)^{1/4} \tag{4}$$

where ρ density of polymer solution, Q volumetric flow rate of polymer solution from the nozzle, $U = U(z)$ potential of a point at z distance from nozzle tip and I electrical current caried by the jet. If Eq. (4) is re-arranged by considering the electrical potential at a distance of point z from nozzle tip in terms of electrical field intensity $U = E \cdot z$, then radius of jet can be described as:

$$r_f = \left(\frac{\rho Q^3}{2\pi^2 IE \cdot z}\right)^{1/4} \tag{5}$$

On the other hand, the intensity of electrical current due to the polymer jet flow can be described as below [38]:

$$I = kE(\pi\gamma\varepsilon_0 r_0 Q)^{\frac{1}{2}} \tag{6}$$

where k is a dimensionless constant ($k \sim 2-6$), E homogeneous electrical current intensity, γ electrical conductivity of polymer solution, ε_0 dielectric constant of space, r_0 radius of nozzle.

By constituting current intensity in Eq. (6), then a relation between jet radius and other technical parameters can be obtained as below:

$$r_f = \left(\frac{\rho Q^{5/2}}{2k\pi^{5/2}(\gamma\varepsilon_0 r_0)^{1/2}\, E^2 \cdot z}\right)^{1/4} \tag{7}$$

If Eq. (7) is analysed further, it can be seen that radius of nanofibers collected onto a collector can change theoreticially by E, as $r_f \sim E^{-0.5}$ by assuming other parameters are constant.

To verify this approach; let us consider the distance between nozzle and collector $L1 = 19.5cm$ when we use collector thickness as 5 mm and $L2 = 19cm$ when we use collector distance as 10 mm, regarding applied potential as V=18kV, we obtain electrical field intensity as $E1 = U/L1 = 0.923\ kV/cm$ and $E2 = 0.947kV/cm$, respectively. In this case, it can be obtained as $(E_2/E_1)^{0.5} = 1.013$. If we consider the relations of $r_{f1} \sim E_1^{-0.5}$ ve $r_{f2} \sim E_2^{-0.5}$ as described above Regarding Equation (7), then we obtain $r_{f1}/r_{f2} \approx (E_2/E_1)^{0.5}$. Finally if this values are compared by experimental results given by Table 7, then it can be clearly seen that:

$r_{f1}/r_{f2} \approx 565.93 \pm 13.49/548.53 \pm 15.45 \approx (E_2/E_1)^{0.5}$ (regarding experimental results with Cu collector)

$r_{f1}/r_{f2} \approx 626.91 \pm 16.75/618.50 \pm 16.49 \approx (E_2/E_1)^{0.5}$ (regarding experimental results with Al collector)

Consequently, the analysis in this part shows that experimental results given in Part 3 is in a good agreement with such a theoretical approach. This analysis also shows that the distance between nozzle and collector distance has critical importance and should be arranged at micrometer level in electrospinning.

4.2. Theoretical analysis regarding fiber radius when different collector material is used

As collection of nanofibers onto a collector has been carried out by an electrical field, the physical properties of particles that constitute nanofiber (such as conductivity, dielectric constant, specific density, viscosity) has primer importance. In this case, particle and collector electrode can be considered as condensator charged at U voltage value:

$$U = U_p - U_0 \tag{8}$$

where U_p potential of particle, U_0 potential of collector electrode which will be zero in our case.

When nanofibers contact surface of collector electrode, the discharge speed of electrical charge of particles in a fiber can be described as below:

$$\frac{dq}{dt} = -\frac{U}{(R_v+R_s+R_c)} e^{-\frac{t}{C\cdot(R_v+R_s+R_c)}} = -\frac{\left(1+2\frac{\varepsilon-1}{\varepsilon+2}\right)E\cdot r_p^2}{C\cdot(R_v+R_s+R_c)} \tag{9}$$

where C is capacitance of fiber-collector system, R_v is volumetric resistance of particle, R_s is surface density of particle and R_c is contact resistance of particle.

The Eq. (9) shows that the change in speed of charge on fibers has been significantly depend on conductivity of collector electrode material when other technical parameters are constant in electrospinning. This relation shows that as conductivity of collector material gets higher, discharge of fibers that were electrically charged would be faster and as a result of this, Coulomb interaction force value between particles constituting nanofiber would get weaker. In other words, when a collector having a higher electrical conductivity is used, the electrical charges have been transferred to the collector much faster and this would lead to reduction in mean fiber radius as a result.

To verify the contribution of this assumption, the experimental results obtained by use of two different collector electrodes (Al and Cu) which have two different electrical conductivitiy has been considered. The experimental results show that average diameter of fibers obtained by use of an collector made of Cu is finer compare to those obtained by use of collector made of Al supporting the explanation above. Furthermore, the above theoretical approach also show that when a collector made of higher electrical conductivity is used, the electrical charges have been transferred to collector much faster and there would be less repulsive force between fibers. Such a less repulsive force would lead less scattering area of fibers collected on collector. The Figures 2 and 3 showing area of collection of fibers on the collector also confirms this theoretical approach as collected area of fibers are distributed at much less area when Cu collector is used compare to the Al collector.

5. CONCLUSION

In this work, the effect of collector plate thickness and collector material on nanofiber fineness was analyzed by producing electrospun nanofibrous surfaces as well as analyzing visually the collection area of fibers on these different collector types. For this purpose, aluminum, and copper collectors with a thickness of 1 mm, 5 mm, 10 mm in circular form were used. The change in fiber fineness when different collector thickness and collector material used has also been analyzed with a theoretical approach as well.

The experimental findings show that finer fibers are obtained with both aluminum and copper collectors because of the increase in metal collector thickness. The difference between fiber fineness was significant when using aluminum collectors with 5 mm and 1 mm thicknesses while it was not statistically significant when aluminum collectors with 10 mm and 5 mm thicknesses were used. On the other hand, a statistically significant difference was observed in terms of the fineness of the fibers when 10 mm and 5 mm thick copper collectors were used. It is thought that finer nanofibers are obtained with much higher elongation of the jets because of the electrical conductivity increasing with the increase of the metal collector thickness. At the same time, it was observed that with the decrease of the metal collector thickness, the nanofibers were dispersed and collected at a larger area on the collector. Therefore, it can be said that the thickness of the collector metal affects morphological properties of nanofibrous surfaces produced by electrospinning such as fiber fineness and fiber collection area, therefore interfiber spacing.

The experimental results show that nanofibers obtained by using copper collector plate are finer than the nanofibers obtained with aluminum collector and their difference is statistically significant. Producing finer fibers with copper collector also explained theoretically as conductivity of collector material gets higher, the electrical charges have been transferred to the collector much faster and this would lead to reduction in mean fiber radius as a result. At the same time, it was observed that the fibers were dispersed at a smaller area on the surface of copper collector compared to the aluminum collector leading to production of relatively tightly packed nanofibrous surface. This was also explained theoretically as the electrical charges have been transferred to the collector much faster and there would be less repulsive force between fibers leading to less scattering area of fibers collected on the collector when a collector made of higher conductivity material is used.

The findings of this work show that a refinement in electrospinning set-up can be carried out by correct choice of collector thickness and material.

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