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Copper-Electroplating of Biodegradable PCL Nanofiber Mats

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ABSTRACT

In this study, biodegradable polycaprolactone (PCL) nanofibers were copper (Cu) electroplated in a more environmentally friendly bath compared to conventional electroplating baths. The Cu-plating mechanism and determination of the optimum parameters for the production of Cu-plated PCL nanofiber mats were explained. PCL nanofibers were produced on metal frames by electrospinning. Cu-electroplating needs a conductive surface. To provide this, a gold/palladium (Au/Pd) mixing was sputtered on the PCL samples with different sputtering thicknesses (1-5-10-15 and 20 nm). After determining the minimum sputtering thickness as 5 nm, the samples were Cu-plated for 1, 3, 5, and 30 minutes in a citric acid electroplating bath. The surface properties of the samples were evaluated after Au/Pd sputtering and electroplating, respectively. Elemental analyses, mapping, and electrical characterizations were also performed after electroplating. After Au/Pd sputtering, the SEM images showed that randomly aligned nanofibers with an average diameter of 223 nm were produced. After electroplating, the average nanofiber diameters increased up to 444 nm. It was seen that the coating grew along the surface of the single nanofibers indicating a smooth Cu coating. While elemental analyses presented a Cu content of 79.77%, electrical characterizations gave a sheet resistance value of 5.98 mΩ/sq for the samples Cu-plated for 30 minutes, indicating a highly conductive structure. Every step of the study is described in detail to provide insight for further studies.

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KEYWORDS

Nanofiber, Electrospinning, Biodegradable Electronics, Copper Electroplating, Electrical Characterization

1. INTRODUCTION

With the spectacular developments in technology, the use of electronic devices has tremendously increased, and they have become an indispensable part of human life. Although our lives have become much easier and more comfortable by the fast emergent technology, the rapid developments have led to the shortened lifetime of electronics resulting in a growing ecological problem such as electronic waste management. In order to reduce these electronic wastes and toxic residues, more environmentally friendly production methods and electronic devices should be developed. This drives the search for an alternative to currently used materials: biodegradable, safe, and non-toxic materials for biodegradable electronics [1–3].

Polycaprolactone (PCL) is a semi-crystalline linear aliphatic polyester that shows good mechanical properties besides its biodegradability and non-toxic nature. It is also a promising material for biodegradable electronics due to the ester bond, which imparts hydrolytic degradation. Moreover, it can be cost-effectively produced from renewable sources, making them more preferable for such applications than other biodegradable polymers [4].

Apart from biodegradability, a lightweight and flexible substrate with a high surface area per volume is needed for an electronic device. Nanofibers meet these expectations perfectly with their small diameters, small pore sizes, and

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high porosity. Electrospinning is the most widely used method for producing nanofibers with high surface area per volume, high porosity, and small pore size [5–8]. It is a relatively simple and fast method that allows nanofiber production from many polymers. One of the most important advantages of electrospinning is producing nanofibers with desirable properties for advanced applications through combining more than one material or method together.

Electrically conductive nanofibers have gained attention in numerous applications, such as wearable electronics, sensors, heaters, solar cells, supercapacitors, and so on [9–15]. These structures provide appropriate properties for applications where electrical conductivity is needed. The decrease in the diameter contributes to the increasing electrical properties due to the confinement [16]. Besides, their higher surface area to volume ratio is also favorable for such applications.

There are different approaches to produce electrically conductive nanofibers by electrospinning, including using an intrinsically conductive polymer and adding a conductive component into the nanofiber. Another approach is coating a non-conductive nanofiber mat (template nanofiber mat) with a metal [17].

Electroplating is one of the most common methods in the deposition of metals on surfaces. In this process, conductive or semi-conductive materials are coated with a thin metal layer in a bath containing suitable chemicals [18-20]. However, conventional electroplating baths contain many toxic and non-environmentally friendly chemicals. Three types of solutions are in common use for copper electroplating. These are acidic (sulfate), alkaline (cyanide and—more seldom—non-cyanide), and pyrophosphate (mildly alkaline) baths. One of the most common chemicals in acidic electroplating solutions is sulfuric acid. Potassium pyrophosphate, potassium o-phosphate, copper cyanide, and sodium cyanide are the chemicals used in alkaline and mildly alkaline electroplating baths. Cyanide solutions have been widely used in the plating industry; however, due to the numerous environmental and occupational hazards, their use has steadily decreased since the 1970s. Acidic copper baths are not expensive; they are simple in composition, stable, and easy to control [21]. While sulfuric acid is commonly used, it is a corrosive substance, destructive to the skin, eyes, teeth, and lungs. Severe exposure can result in death [22].

On the other hand, electroplating in a citric acid bath is also applicable [23-28]. Citric acid is one of the most widely used organic acids. The U.S. Food and Drug Administration (FDA) states that citric acid is generally recognized as safe (GRAS) as a direct food additive [29]. It is used as an acidulant and preservative in the food industries and also used as a complexing agent and starting material for functional compounds in the pharmaceutical and cosmetic industries. Citric acid is also used as a complexing and chelating agent in metal treatment, as a water softener for detergents, and as a buffering agent in the toiletry and pharmaceutical industries [30].

In the nanofiber electroplating process, the nanofiber mat is used as the cathode, and the metal to be coated on the nanofiber is used as the anode (Figure 1). Afterwards, the anode and cathode are placed in the electrolyte solution, and a voltage is applied. The metal ions begin to move towards the cathode, and the nanofiber mat is coated with the metal. Many metals, including copper (Cu), nickel (Ni), silver (Ag), and gold (Au), can be used for the electroplating process. Among them, Cu is preferable, especially for its high electrical conductivity, non-toxicity, and availability.

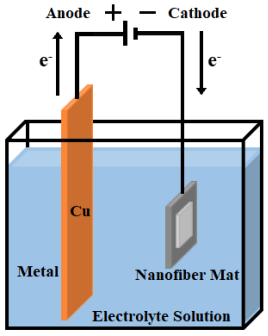


Figure 1. A schematic view of the electroplating of nanofibers

Although electroplating is a well-known and used method in many applications, studies on the electroplating of nanofibrous surfaces are rare. Besides, most of these studies have focused on the electroplating of non-biodegradable PAN and PU nanofiber mats in conventional electroplating baths [20,31-37].

The aim of this study is to produce highly conductive PCL nanofibers with sheet resistance values in the range of milliohms. In this respect, Cu-plated PCL nanofiber mats were produced with the combination of electrospinning and electroplating. Another focus of this study is to understand and explain the Cu-electroplating mechanism and determine the optimum parameters for the electroplating process of biodegradable electrospun nanofiber mats. PCL nanofibers were produced by electrospinning to fulfill the aforementioned condition. A critical point in the electroplating process is that the nanofiber mats should be slightly conductive to be metal-coated in the electroplating solution. Therefore, the nanofiber mats are usually coated by a thin conductive layer prior to electroplating. For this purpose, PCL mats were coated by Au/Pd with different sputter coating thicknesses. The minimum thickness was selected for the Cu electroplating to avoid the excess usage of Au/Pd with environmentally friendly respect. One of the distinctive points of this study is that Cu-electroplating was achieved using a bath containing more environmentally friendly chemicals such as citric acid compared to the conventional electroplating baths containing sulfuric acid or cyanide.

In order to understand the Cu electroplating mechanism, different durations (1-3-5-30 min) were applied for the Cu electroplating on the samples. The nanofiber mats were characterized in terms of surface and electrical properties after electrospinning, sputter coating, and electroplating processes, respectively. Also, elemental analysis and mapping were studied to reveal atomic compositions and homogeneous distribution of Cu on the surface of prepared samples.

2. MATERIALS AND METHODS

2.1 Materials

In this study, PCL (Mn:80000 Da, Sigma Aldrich) was used as the polymer, dichloromethane (DCM), and N,N-Dimethylformamide (DMF) were used as the solvents. Copper sulfate, citric acid, and distilled water were used for the electroplating solution [23]. All the chemicals were used without further purification.

2.2 Methods

The experimental procedure followed in the study is given in Figure 2.

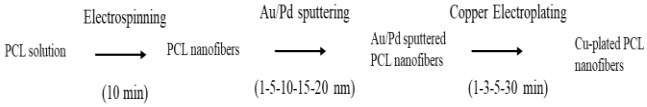


Figure 2. The experimental procedure followed in the study

Solution preparation: In this study, 10% wt. of PCL polymer was dissolved in a DMF/DCM (60:40) solvent system. The solution was kept on a hot plate at 40 °C until it was completely dissolved.

Electrospinning of PCL solutions: An Inovenso electrospinning device was used to produce PCL nanofibers. The device consists of a feeding pump, a high voltage source, and a rotating drum collector. The electrospinning parameters were selected as a flow rate of 0.7 ml/h, a voltage of 19 k,V and a distance of 15 cm. The fibers were collected on metal frames (internal dimensions: 1 x 1.5 cm) mounted on the drum collector rotating at a speed of 300 rpm for 10 minutes (Figure 3).

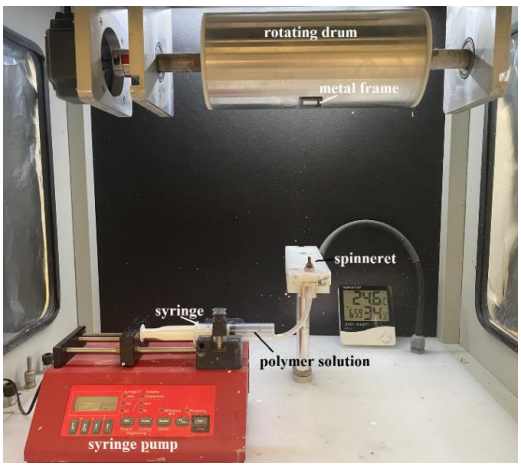


Figure 3. The electrospinning set-up used in the study

During electroplating, the nanofibers on the metal frame had a tendency to flow into the electroplating solution. In order to prevent this, another frame was attached on the nanofiber mat, and the nanofiber mat was fixed between these two frames (Figure 4).

Au/Pd Sputtering of PCL nanofibers: In order to conduct electroplating, the nanofiber mats should be slightly conductive. Therefore, PCL nanofiber mats were Au/Pd-sputtered by a Leica EM ACE600 model sputtering system before electroplating. The minimum thickness of the coating was determined by preliminary studies to avoid excess usage of Au/Pd. The nanofiber mats were Au/Pd sputtered with different thicknesses (1-5-10-15-20 nm) (Figure 5). Afterwards, electroplating was performed on each sample, and the minimum thickness that allows a homogenous coating in electroplating was determined as 5 nm for this study.

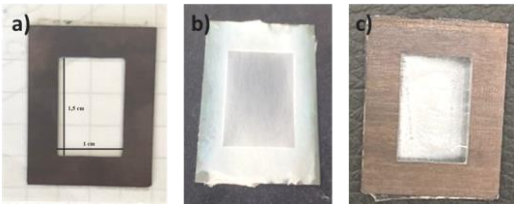


Figure 4. The nanofiber deposition procedure on the metal frames: **a)** The designed metal frame, **b)** The nanofibers deposited on the metal frame, **c)** The nanofiber mat fixed between two metal frames

(a) (b) (c) (d) (e)

Figure 5. Au/Pd sputtered nanofiber mats with different sputtering thicknesses: **a)** 1 nm, **b)** 5nm, **c)** 10 nm, **d)** 15 nm, and **e)** 20 nm, respectively

Cu electroplating of PCL nanofibers: After sputtering, the nanofiber mats were immersed in an electroplating solution. For the electroplating solution, 100 g copper sulfate and 50 g citric acid were added into the 700 mL distilled water. A pure Cu plate was used as the anode, and the nanofiber mat deposited on the metal frame was used as the cathode. In electroplating, Cu ions (Cu^{2+} and Cu^{+}) are released from the anode and attaches to the nanofiber mat. These reaction mechanisms are given in Equations 1 and 2, respectively [31].

$$Cu \rightarrow 2e^- + Cu^{2+} \text{ or } Cu \rightarrow e^- + Cu^{+} \quad (1)$$

$$Cu^{2+} \rightarrow 2e^- + Cu \text{ or } Cu^{+} \rightarrow e^- + Cu^{+} + e^- \rightarrow Cu^{+} \quad (2)$$

For a homogenous coating, other important parameters are voltage and current. These values should be selected carefully. In conventional electroplating baths, current density differs between 20-100 mA/cm^2 [21]. In an ideal electroplating process, it is aimed to properly coat the material and complete the process as soon as possible. However, these two parameters are opposite to each other. In order to accelerate the process, the amount of current needs to be increased. On the hand, the increase in the current results in the deterioration of the coating quality, which is undesirable. In this study, the aim was also to obtain smooth coatings on nanofibers. Therefore, many trials were conducted, and optimum voltage and current values were determined. When the current was very low, the coating process was prolonged. On the other hand, when the current was increased to very high values, the metal frame was coated very fast, and the coating on the nanofibers were deteriorated. Consequently, the optimum parameters for a better coating were determined as 3 V and 0.5 A. All the samples used in the study were Cu-plated with these values for 1-3-5 and 30 minutes (Figure 6). After electroplating, the samples were rinsed with distilled water.

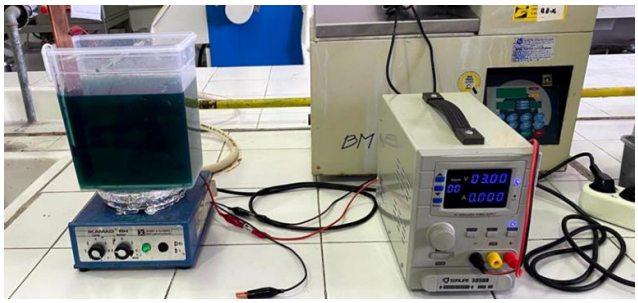


Figure 6. Electroplating set-up used in the study

Characterization of the nanofiber mats: A digital camera was used for the visual evaluation of the samples after electrospinning, Au/Pd sputtering, and electroplating by placing the samples over the logo.

The surface morphology of the samples before electroplating was investigated by a Carl Zeiss Evo 40 scanning electron microscope (SEM). The SEM analyses were also used to investigate the surface morphologies and the elemental analysis of the samples after electroplating by A Carl Zeiss/Gemini 300 SEM. The nanofiber diameters were calculated using Image J software. The thicknesses of PCL nanofiber mats were measured with an electronic digital micrometer before and after the electroplating process. An FPP 470-Entek Electronic four-point device was used to determine the electrical properties of the samples.

3. RESULTS AND DISCUSSION

Figure 7 shows the SEM image of PCL nanofibers at 5 nm Au/Pd sputtering before electroplating. It can be seen that randomly aligned nanofibers with uniform diameters around 223 nm were produced.

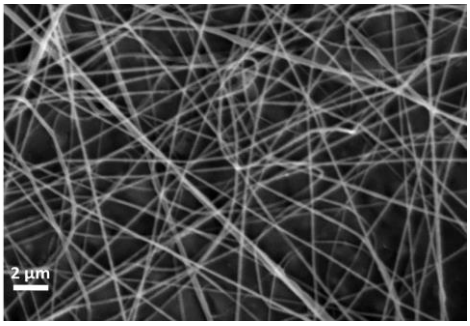


Figure 7. SEM image of the PCL nanofibers at 5 nm Au/Pd sputtering before electroplating

Figure 8 shows the visual evaluation of the nanofibers after Cu-electroplating with different durations (1, 3, 5, and 30 min). After 1 and 3 minutes of electroplating, no visible Cu coating was observed on the nanofiber mats. After 5 minutes, Cu coating started to appear from the edges of the metal frames. Cu almost fully covered the surface of the sample at 30 minutes of electroplating. The nanofiber mat thickness values increased from 22.40 µm to 95.60 µm on average after 30 minutes of electroplating.

(a) (b) (c) (d)

Figure 8. Visual evaluation of nanofibers after: **a)** 1 min, **b)** 3 min, **c)** 5 min, and **d)** 30 min of electroplating

The Cu coating mechanism of the nanofiber mats can be understood more deeply by SEM analyses. The coating grew along the surface of the single nanofibers. The nanofiber diameters increased up to 444 nm due to the coating (Figure 9).

Elemental analyses were performed from two different areas; uncoated and Cu-coated. A Cu content of 79.77% was observed on the coated area, while content of 0.81% was detected on the uncoated section (Figure 10). In Figure 11, the red-colored area shows the Cu presence, which was only observed in the coated parts of the surface. This confirms the results of the elemental analyses.

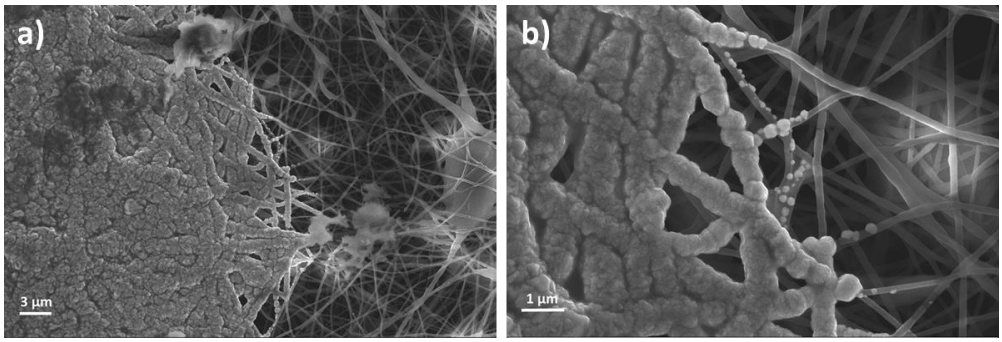
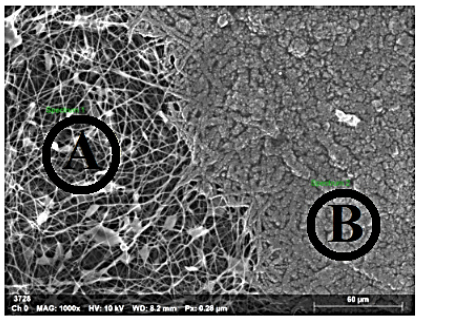


Figure 9. SEM images of 30 min Cu-plated nanofibers with a magnification of: **a)** x2500, **b)** x10000



SEM-EDS Area	Carbon (%)	Oxygen (%)	Copper (%)
A	72.15	27.04	0.81
B	11.74	8.49	79.77

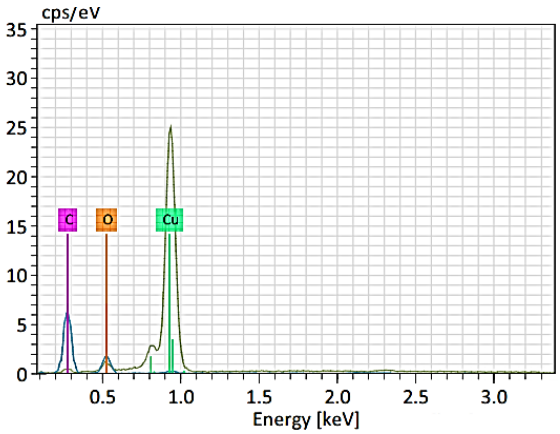


Figure 10. Elemental analyses result of the Cu-plated nanofiber mats

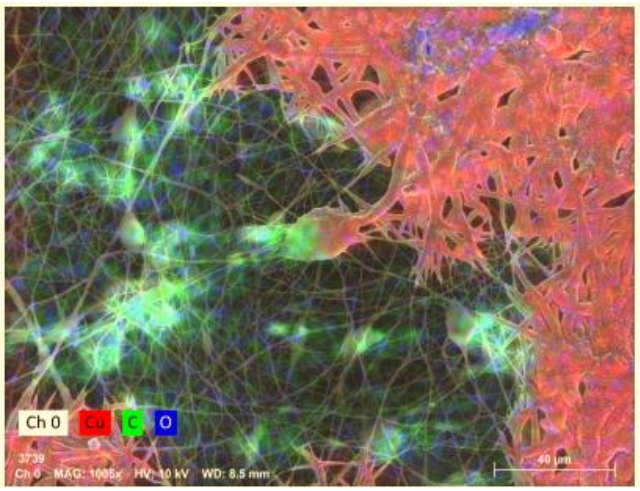


Figure 11. Mapping image of the Cu-plated nanofiber mats

Depending on the duration of the electroplating process, the Cu partially covered the nanofiber mat surface. For this reason, the sheet resistance values were measured from both uncoated and Cu-coated areas. The uncoated areas gave sheet resistance values in the range of megaohms, which was quite high. Since there was no Cu present on the uncoated areas, these values could be considered as the sheet resistance values of the Au/Pd sputtered areas. However, it is not possible to perform electroplating at such high resistance values. This unexpected sheet resistance value is most probably due to the contact of the measurement probe with the non-sputtered nanofibers underneath the surface. The same trend was also seen for the Au/Pd sputtered samples before electroplating. On the other hand, Cu-plated areas had sheet resistance values in the range of milliohms, meaning that these surfaces were highly conductive (Table 1).

Table 1. Electrical characterization of the Cu-plated nanofibers with different electroplating times

	Rs (mΩ/sq)						
	1 min ep		3 min ep		5 min ep		30 min ep
Before Electroplating (5 nm Au/Pd sputtering)	Non-coated area	Cu-plated area	Non-coated area	Cu-plated area	Non-coated area	Cu-plated area	Cu-plated area
0.34×10^9	0.42×10^9	-	0.33×10^9	-	0.41×10^9	7.34	5.98

4. CONCLUSION

With their high surface area, lightweight, good mechanical properties, and biodegradable nature, PCL nanofibers stand out as alternative substrates for the production of biodegradable electronics. In order to prepare appropriate surfaces for such applications, their surface can be metalized by electroplating. However, the electroplating of nanofibrous materials can be challenging since they are thin and delicate. The aim of this study is to explain the electroplating process of this kind of unique structures in detail. Moreover, in this study, a more environmentally friendly electroplating solution is preferred compared to conventional solutions.

The study carried out can be summarized as follows:

- Special metal frames were designed to handle the nanofiber mats in the electroplating bath.
- Biodegradable PCL nanofibers were electrospun successfully on the metal frames.
- The minimum Au/Pd sputtering thickness was determined as 5 nm for this study.
- The optimum voltage and current for a homogenous electroplating process were evaluated.
- Electroplating was performed on nanofiber mats with four different durations.
- Cu coating started to appear from the edges of the metal frames and grew on the single nanofiber surface.
- Elemental and mapping analyses confirmed the presence of Cu on the nanofiber mats.
- Low sheet resistance values in the range of milliohms were obtained on the Cu-coated areas.

It can be concluded that Cu-plated PCL nanofiber surfaces can be produced by combining electrospinning and electroplating. These kinds of structures hold great potential for biodegradable electronics due to their high conductivity and biodegradability.

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