Polyurethane Nanofiber Polymer: preparation, production, characterization and properties

Polímero de Nanofibras de Poliuretano: prensa, produzão, caracterização e propriedades

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ABSTRACT
The present article provides an overview of polyurethane (PU) nanofibers (NFs), focusing on the process of fabrication of electrospun PU-NFs using the electrospinning method. The electrospinning device is used to produce PU by changing three main parameters in order to control the shape and thickness of the fibers, as well as the presence of bubble-like structures in the produced fibers. The first parameter is the PU concentration (8% and 10%), but this requires knowledge of the concentrations of the solvents (DMF: dimethylformamide, and THF: tetrahydrofuran). The second parameter is the flow rate of the dissolved PU from the syringe needle (0.02 and 0.03 mL/min), and the third parameter is the distance between the needle tip and the metal plate of the electrospinning device (7, 10, and 15 cm). These parameters were calibrated to obtain a sample with the desired strength and pressure characteristics. The obtained PU fibers are characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR). The results show that 10% of PU fibers are in the nanoscale range in diameter and present a typical FTIR PU profile. Two concentrations were studied (8% and 10%), but only 10% systems gave satisfactory results. Because of their excellent mechanical properties, electrospun PU-NFs can be used in a wide range of applications, including high-performance air filters, wound dressing materials, protective textiles, and much more.
Keywords: electrospinning, polyurethane, nanofibers, characterization.

RESUMO
O presente artigo fornece uma visão geral das nanofibras (NFs) de poliuretano (PU), com foco no processo de fabricação de PU-NFs eletrospun usando o método de electrospinning. O dispositivo de electrospinning é utilizado para produzir PU alterando três parâmetros principais para controlar a forma e a espessura das fibras, bem como a presença de estruturas semelhantes a bolhas nas fibras produzidas. O primeiro parâmetro é a concentração de PU (8% e 10%), mas isso requer conhecimento das concentrações do solvente (DMF: dimetilformamida e THF: tetrahidrofurano). O segundo parâmetro é a vazão do PU dissolvido da agulha da seringa (0,02 e 0,03 mL/min), e o terceiro parâmetro é a distância entre a ponta da agulha e a placametálica do dispositivo de electrospinning (7, 10 e 15 cm). Esses parâmetros foram calibrados para obter uma amostra com as características de resistência e pressão desejadas. As fibras de PU obtidas são caracterizadas por microscopia eletrônica de varredura (MEV), difração de raios X (XRD) e espectroscopia no infravermelho com transformada de Fourier (FTIR). Os resultados mostram que 10% das fibras de PU estão na faixa da nanoescala em diâmetro e apresentam um perfil típico de PU FTIR. Foram estudadas duas concentrações (8% e 10%), mas apenas os sistemas de 10% deram resultados satisfatórios. Devido às suas excelentes propriedades mecânicas, os PU-NFs eletrospun podem ser utilizados em uma ampla gama de aplicações, incluindo filtros de ar de alto desempenho, materiais para curativos, tecidos protectores e muito mais.

Palavras-chave: electrospinning, poliuretano, nanofibras, caracterização.

RESUMEN
El presente artículo proporciona una visión general de las nanofibras de poliuretano (PU) (NFs), centrándose en el proceso de fabricación de PU-NFs electrospun utilizando el método de electrospinning. El dispositivo electrospinning se utiliza para producir PU cambiando tres parámetros principales para controlar la forma y el grosor de las fibras, así como la presencia de estructuras similares a burbujas en las fibras producidas. El primer parámetro es la concentración de PU (8% y 10%), pero esto requiere conocer las concentraciones del disolvente (DMF: dimetilformamida y THF: tetrahidrofurano). El segundo parámetro es la velocidad de flujo de la PU disuelta de la aguja de la jeringa (0,02 y 0,03 ml/min), y el tercer parámetro es la distancia entre la punta de la aguja y la placa metálica del dispositivo de electrohilado (7, 10 y 15 cm). Estos parámetros se calibraron para obtener una muestra con las características de resistencia y presión deseadas. Las fibras de PU obtenidas se caracterizan utilizando microscopía electrónica de barrido (SEM), difracción de rayos X (XRD) y espectroscopia infrarroja de transformada de Fourier (FTIR). Los resultados muestran que el 10% de las fibras de PU están en el rango de nanoescala en diámetro y presentan un perfil típico de PU FTIR. Se estudiaron dos concentraciones (8% y 10%), pero solo el 10% de los sistemas dieron resultados satisfactorios. Debido a sus excelentes propiedades mecánicas, los PU-NF electrospun pueden utilizar en una amplia gama de aplicaciones, incluyendo filtros de aire de alto rendimiento, materiales para heridas, textiles protectores y mucho más.

Palabras clave: electrospinning, poliuretano, nanofibras, caracterización.
1 INTRODUCTION

Progress in fiber science in the last quarter of the 20th century focused on the development of polymer fibers. Specifically, nanofiber technology has become a growing trend and has contributed to many inventions for various textile applications[1].

Nanofibers are widely used in regenerative medicine and tissue engineering. Electrospinning is a method used to produce and fabricate nanofibers, and it can be said that it is one of the most simple and convenient ways to manufacture them[2][3].

Electrospinning is a quick and controllable process for generating a fine fiber from a variety of materials that may include a polymer. The electrospinning device consists of three main parts, which are a high-voltage power supply, a syringe with a metal needle, and a conductive collector.

Polyurethane (PU) has received significant attention in electrospinning process applications for this new generation of materials. Some researchers have focused on the production of nanofibers from different polymers via the electrospinning process [4][5]. Polyurethane polymer compounds are widely used as biomaterials due to their excellent mechanical and water-insoluble properties. The use of the electrospinning method to produce polyurethane nanofibers will result in a porous network structure and a large surface area. Therefore, electrospun polyurethane nanofibers can be used in many aspects of our daily lives, for instance, in building, textiles, furniture, acoustic and thermal insulation, shoes, automobiles, electronics, and biomedical equipment[6]. The polymer solution properties are the most influential factors in the electrospinning procedure and the resultant nanofiber structure.

The history of electrospinning begins in 1914, when John Zeleny was working to treat what is called the end of iron capillaries in a liquid drop. While he was doing his research, he tried to generate a mathematical model for liquids under the conditions of electrostatic forces [6].

In the 1930s, Formhals made the first attempt at producing electrospun fibers. But his method was having some issues, for instance, the drying of the fibers (due to the long distance between the collector and the nozzle)[7][8].

Later on, in 1940, he improved his system to be more reliable. Between 1964 and 1969, Sir Geoffrey Taylor established the basis for the theoretical electrospinning method. His work led to the modeling of the hopper, in which the electric field attracts the liquid drops[9][10][11][12]. Later on, his collaboration with JR Melcher helped to upgrade the "leaky dielectric model".
The electrospinning process can successfully and cost-effectively make fibers, and this is why it got a lot of attention in the scientific community, especially because these fibers range in diameter from a few micrometers down to just tens of nanometers. In this process, a solvent containing the dissolved polymer is forced to become a nanometer-scale fiber with the help of a strong electric field. This happens when the strength of the applied electric field becomes greater than the surface tension of the drop of the liquid polymer; now a solution jet has been formed and will be accelerated toward the collector electrode (the opposite electrode). The solution will be dry when it reaches the second electrode by air, and the nanofibers will be formed and collected on a surface (the collection zone). The resulting nanofiber fabrics resemble the structure of a natural extracellular matrix, and that is the reason why these nanofibers have gained growing interest in drug delivery, tissue engineering, and biomedical applications.[13][14].

The fibers fabricated by the electrospinning process have small inter-fibrous pore sizes, high porosity, diameters on the nanometer scale, vast possibilities for implementation with a high surface area to volume or mass ratio, adaptability, ease of use, and machine setup. Due to these unique properties, it gained a lot of attention. Using this technique, various polymers have been successfully fabricated and electrospun to make ultrafine fibers, which include polyurethane (PU)[15][16].

This work aims to have polyurethane nanofibers intended for use in various biomedical applications. Therefore, two samples of PU were prepared with two different concentrations, the first one with a concentration of 8% and the other one with a 10% concentration, using an electrospinning device. The resultant PU composite nanofibers were analyzed by scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR).

2 EXPERIMENTAL Results AND DISCUSSION

Granulated polyurethane (PU) was purchased from Termosan (Istanbul, Turkey). Tetrahydrofuran (THF) and dimethylformamide (DMF) were obtained from Sigma Aldrich (St. Louis, MO, USA).

PU preparation parameters:

As the solvent type is directly related to the electrospinning solution properties, the type of solvent and its concentration affect the morphological structure of the electrospun nanofiber matrix significantly. The solvents that are usually used to dissolve PU are all highly polar organic solvents, for instance, N-methylpyrrolidone, N-dimethylformamide,
and tetrahydrofuran (THF). With the solvent selection, it was found that the morphology of the resultant nanofibers changed significantly [14].

Preparation of 8% and 10% polyurethane was conducted using a mixture of the following solvents: dimethylformamide; DMF (molar mass = 73.095 g.mol⁻¹, density d=0.948), and tetrahydrofuran; THF (molar mass = 72.11 g.mol⁻¹, density d=0.889). 8% then 10% of PU were prepared in 20 ml of solvent (10 ml DMF + 10 ml THF). The following table shows the results of the preparation:

<table>
<thead>
<tr>
<th></th>
<th>8% →100 ml</th>
<th>10% →100 ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mₚᵤ→20 ml</td>
<td>Mₚᵤ→20 ml</td>
<td></td>
</tr>
<tr>
<td>Mₚᵤ=1.6 g</td>
<td>Mₚᵤ=2 g</td>
<td></td>
</tr>
</tbody>
</table>

Table 1: Preparation results of 8% and 10% PU samples.

Source: The authors, 2024

The parameters of the preparation were:

- The PU concentration ranged between 8 and 10%.
- The applied voltage was 15–30 kV.
- The tip-to-collector distance was 7–15 cm.
- The feeding or flow rate was set at 0.02 mL/min and 0.03 mL/min.

2.1 SOLUTION PARAMETERS

2.1.1 Concentration

The formation of nanofibers is largely dependent on the solution's concentration. A minimum concentration of spinnability exists. The entangled polymer will detangle before reaching the collecting device when the solution concentration is lower than this value due to the low concentration and high surface tension. This is caused by the interplay between the electric field force and surface tension[17].

2.1.2 Viscosity

One of the primary factors influencing the diameter and morphology of nanofibers is the concentration and molecular weight, which are closely correlated with the solution's viscosity. A bead structure will occur if the viscosity is too high or too low. When the viscosity is excessively low, the polymer Surface tension predominates and entanglement is minimal[18].
The droplets are unable to create spray by attaching to fibers. Viscosity increases cause the polymer's stress relaxation period to lengthen, which promotes the formation of larger, more uniformly sized nanofibers.

2.1.3 Surface tension

Numerous variables, such as molecular weight, solution concentration, solvent type, and temperature, all affect surface tension. Nevertheless, the surface tension can also be successfully lowered by using a surfactant.

which, in the process that follows, just has to be eliminated. The beads are fewer and the nanofibers are smoother and finer when the surface tension is low.

Nanofibers were collected on glass sheets, and the distance between the glass sheets and the tip of the needle was set at different values[19].

2.2 PROCESSING PARAMETERS

2.2.1 Applied voltage

Another important factor in the electrospinning process is the applied voltage. The droplet cannot be expelled and will not ultimately reach the collection device until the critical voltage is reached. The diameter and shape of the nanofibers are also impacted by the applied voltage. Beads will occur if the applied voltage is either too high or too low[20].

2.2.2 Flow rate

The diameter and form of nanofibers are also influenced by the flow rate. As the flow rate increases, so does the diameter of the nanofibers.

Beads or ribbons will form if the flow rate is too high because the solvent will not be able to evaporate entirely before it reaches the collection device. As a result, the Taylor cone is more stable and producing smooth, homogeneous nanofibers is simpler at moderate flow rates. According to some experts, there is an ideal flow rate, and when it is not reached, nanofibers get coarser[21][22][23].
2.2.3 Optical microscope

Tables 2 and 3 illustrate the most comprehensive results obtained by optical microscopy (optical microscope type Olympus AnalySIS, USA) for 8% and 10% PU, respectively.

It is noted that with 8% PU samples, no fibers were obtained or the fibers were unclear. So, in this section, only results concerning 10% PU samples are reported.

Table 2: 8% PU images obtained using an optical microscope.

<table>
<thead>
<tr>
<th>Table A</th>
<th>Distance 10 cm</th>
<th>Flow rate 0.02 ml/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension (kV)</td>
<td>15 kv</td>
<td>18 kv</td>
</tr>
<tr>
<td>Picture</td>
<td>20 kv</td>
<td>25 kv</td>
</tr>
<tr>
<td>Tension (kV)</td>
<td>18 kv</td>
<td>20 kv</td>
</tr>
<tr>
<td>Picture</td>
<td>25 kv</td>
<td>30 kv</td>
</tr>
<tr>
<td>Tension (kV)</td>
<td>18 kv</td>
<td>20 kv</td>
</tr>
<tr>
<td>Picture</td>
<td>25 kv</td>
<td>30 kv</td>
</tr>
</tbody>
</table>

Source: The authors, 2024
2.2.4 FTIR analysis

The link type in the 10% PU nano-fibers was determined using Fourier transform infrared spectroscopy (FTIR) on a JASCO FT/IR-4000 spectrometer.

The typical FTIR spectrum of a pure PU nanofiber is shown in Figure 1 and 2. The obtained spectrum of pure 10% PU revealed a strong absorption peak of the urethane group’s N-H stretching vibration at 3352 cm\(^{-1}\).

The C–H symmetry of CH\(_2\) groups was observed at 2948 cm\(^{-1}\), and the asymmetric stretching was observed at 2865 cm\(^{-1}\). The other peaks were set as 1365 cm\(^{-1}\) (CH\(_3\) bending vibration), 1525 cm\(^{-1}\) (N–H bending vibration), 1441 cm\(^{-1}\) (CH\(_2\) bending vibration), 1720 cm\(^{-1}\) (C=O bond of the urethane group), 1305 and 1253 cm\(^{-1}\) (asymmetrical and symmetrical C–O stretching vibration of polyol), and 1104 and 1043 cm\(^{-1}\) (asymmetrical and symmetrical C–O stretching vibration of the urethane group)[24].

The following figure represents the FTIR spectrum of pure 10% PU nanofiber:
Figure 1: FTIR spectrum of 10% PU nanofiber.

Source: The authors, 2024

Figure 2: FTIR spectrum of 10% PU nanofiber with wavenumber.

Source: The authors, 2024
In Figure 1 and 2, the importance of the analysis with the FTIR device is shown. The bonds obtained relate only to the polyurethane compound, which confirms the exit of solvent bonds from the polyurethane sample and the purity of the sample.

2.2.5 SEM Observations

The morphology and diameter of the fibers were determined and analyzed using a scanning electron microscope (SEM), the ZEISS EVO MA10, with an applied voltage of 10 kV. Nano-fiber mats are placed on the sample holder of the SEM and were sputter-coated with carbon. The average fiber diameter and the samples' net diameter distribution with different PU concentrations were successfully measured using SEM [16].

This study examined the effect of some parameters on the diameter of the PU nanofibers, including the distance between the syringe tip (nozzle) and the collector plate, the flow rate, as well as the solution concentration. The resultant diameters were calculated from the SEM images, as illustrated in Figure 2. This figure shows the SEM images of the nanofibers obtained when 10 cm was the preset distance between the collector and the tip of the nozzle. It has been observed that the diameters of the nanofibers decrease with increasing distance to the collector. Also, as the solvent can get enough time to evaporate with increasing distance, the distance is controlled to prevent any undesired bead formation[25].

The following images show the SEM images of the 10% PU nanofibers
The importance of the scanning electron microscope is to identify clear images of the nanofiber sample and determine the diameter of the nanofiber through the field specified in biomaterial applications, where the diameter ranges from 150 nm to 400 nm. This is illustrated by the images in Figure 3.

The preparation parameters and their corresponding obtained results (fiber diameter and fiber morphology) are summarized in table 4. Moreover, Table 5 cites some previously reported results for comparison.
Table 4: Polyurethane samples: obtained results and fiber properties.

<table>
<thead>
<tr>
<th>PU</th>
<th>Distance (cm)</th>
<th>Flow rate (ml/min)</th>
<th>Tension (kV)</th>
<th>Fiber diameter (nm)</th>
<th>Fiber morphology</th>
</tr>
</thead>
<tbody>
<tr>
<td>8%</td>
<td>10</td>
<td>0.02</td>
<td>15</td>
<td>No fiber</td>
<td>No fiber</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>0.3</td>
<td>18</td>
<td>No fiber</td>
<td>No fiber</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.02</td>
<td>18</td>
<td>Unclear</td>
<td>Unclear</td>
</tr>
<tr>
<td>10%</td>
<td>10</td>
<td>0.02</td>
<td>18</td>
<td>751.6</td>
<td>Smooth</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>0.02</td>
<td>18</td>
<td>164.3</td>
<td>Smoothest</td>
</tr>
</tbody>
</table>

Source: The authors, 2024

Table 5: Results of previous studies on PU systems.

<table>
<thead>
<tr>
<th>Fiber diameter (nm)</th>
<th>Fiber morphology</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>193</td>
<td>Smooth</td>
<td>[1]</td>
</tr>
<tr>
<td>(1155 ± 161)</td>
<td>diameter uniform</td>
<td>[2]</td>
</tr>
<tr>
<td>483.2</td>
<td>Uneven diameter</td>
<td>[3]</td>
</tr>
<tr>
<td>400</td>
<td>Uneven diameter</td>
<td>[16]</td>
</tr>
<tr>
<td>smoother</td>
<td>Thinner nanofibers.</td>
<td>[17]</td>
</tr>
<tr>
<td>390</td>
<td>Smoothest</td>
<td>[19]</td>
</tr>
</tbody>
</table>

Source: The authors, 2024

2.2.6 XRD Analysis

The structure of the nanofibers was determined using an X-ray diffractometer, type SHIMADZU XRD-6100. The used X-ray tube target is Cu, and the voltage was set to 40 kV with a current of 30 mA. A continuous scan range of 5°–90°, a scan speed of 2°/min, a sampling pitch of 0.02°, and a preset time of 0.6 sec were used.

Phase identification is the first step in X-ray diffraction (XRD) powder analysis, and it is one of the core tasks of the software in HighScore. The identification can be done by the user based on peak positions (relative intensities and d-spacing) and/or based on the experimental pattern's full net profile. In addition, the best results are often obtained using a combination of both methods, which cover both major and minor phases [24].

The ICDD’s Powder Diffraction File (PDF) can be considered a database of organic and inorganic diffraction data that can be used for material characterization and phase identification. The PDF has been available for over 70 years and finds too many applications in X-ray [26].

The ICDD has developed the PDF databases to become chemical analysis tools that can be used with the associated analytical techniques. Developing the tools to identify and quantify materials using known associated analytical techniques was the main driving force for this project [27].
Figure 4 shows the XRD diffractogram of electrospun 10% PU nanofibers. The solvent systems all show a large diffraction peak between 10° and 30°, indicating the presence of a mixed ordered structure with both hard and soft domains and an amorphous 10% PU phase[21]. The peaks at 38.06°, 44.25°, 64.73°, and 77.78° represent the presence of a regular position of the mixed nanofibers; the latter represents a crystalline structure phase of the 10% PU. Which means that the PU nanofiber is obtained by the electrospinning semi-crystalline process[28][29][30].

The following figure represents the 10% PU diffractogram:

![10% PU diffractogram](image)

Source: The authors, 2024

Through analysis using X-ray diffraction device, the structure of polyurethane nanofibre fabric is important. The more regular the nanofibre fabric in the crystalline structure, the wider the scope of its application, especially in biomaterials (eye cornea, ear retina, etc.), as shown in figure 4.
### 2.2.7 Peak list

The following table represents the peak list obtained from HighScore XRD analysis for a 10% PU sample.

<table>
<thead>
<tr>
<th>Position 2θ (°)</th>
<th>Height (cts)</th>
<th>FWHM Left 2θ (°)</th>
<th>d-spacing (Å)</th>
<th>Relative. Intensity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>38.0608</td>
<td>671.91</td>
<td>0.2362</td>
<td>2.36433</td>
<td>87.18</td>
</tr>
<tr>
<td>44.2875</td>
<td>770.74</td>
<td>0.3149</td>
<td>2.0453</td>
<td>100</td>
</tr>
<tr>
<td>64.7302</td>
<td>241.66</td>
<td>0.4723</td>
<td>1.44016</td>
<td>31.35</td>
</tr>
<tr>
<td>77.7804</td>
<td>354.11</td>
<td>0.4723</td>
<td>1.22794</td>
<td>45.94</td>
</tr>
</tbody>
</table>

Source: The authors, 2024

### 2.2.8 DSC Analysis

Testing using differential scanning calorimetry (DSC) was done using a DSC machine (DSC131 Evo). The samples (5.6 mg) were heated at a rate of 10 C°/min from 30 C° to 250 C°. After that, they were maintained at 250 C° for 5 minutes before being cooled to-10 C°at the same pace. Lastly, a second thermal scan was conducted. Every sample's second heating scan result served as a benchmark.

Figures 5 and 6 display the thermograms of a PU 10% sample. Three ranges can be seen by carefully examining the thermal diagram:

**First range**: $t_1=13.2$ min , $t_2=13.8$ min This range represents the absorption of an amount of heat flow (endothermic) estimated at 0.5 (mW), and it also represents The glass transition temperature $T_g$, Which corresponds to a temperature of 153.44 C°, Amorphous polymers transition from a hard, glassy state to a soft, rubbery one, or vice versa, at a temperature known as $T_g$. The $T_g$ has a direct bearing on a material's resistance and suitability for a particular intended use[31][32].

**Second range**: $t_1=22.1$ min , $t_2=22.8$ min This range represents the absorption of an amount of thermal energy (endothermic) estimated at2.153 (J/g), Which corresponds to a melting temperature of $T_f=240.13$ C°.

A polymer's melting temperature ($T_f$) is the point at which it transitions from a rubbery to a liquid or more accurately viscous state. Only the crystalline portion of semi-crystalline polymers undergoes fusion[33][34].

**Third range**: $t_1=34.8$ min , $t_2=38.5$ min This range represents of thermal energy (exothermic) estimated at -4.079 (J/g),Which corresponds to a crystallization temperature of $T_c = 121.96$ C°.
Reliability in determining the temperatures at which crystallization occurs and the energy released is high. It is important to note that during DSC tests, the temperature at which crystallization occurs may shift to lower temperature values. The possibility of supercooling effects during the crystallization process is the cause of this.[35][36][37][38].

Figure 5: Thermogram DSC of heat flow as a function of time for a sample 10% PU.

Figure 6: Thermogram DSC of heat flow as a function of temperature for a sample 10% PU.
Thermal analysis is a conventional analytical method used to determine the authenticity of a polymer. Engineers can test a variety of features of the polymer material, including its physical, electrical, thermal, and optical properties, which makes thermal analysis of polymers crucial. It also clarifies other factors like aging, transitions, the impact of additives, and the way various environmental circumstances affect the polymers.

3 CONCLUSION

The electrospinning process was used to prepare polyurethane nanofibers (PU) at different concentrations of 8% and 10%. The preparatory process of nanoscale samples has been improved by different techniques due to changing the standards of the electrospinning method with homogeneous structures according to different scales in the electrical rotation device, such as liquid flow and dimension between the two panels and latency difference between the two panels, among others. SEM has been used to monitor fiber diameters as the field of use of these fibers requires precision in preparation, especially in biomaterials. The diameter of nanofibers ranges from 150 nm to 400 nm. This has been confirmed by previous research. The assessment of the structure of the nanofibre in the X-ray diffraction device also underscores the importance of the structure of the nanofibre fabric. This was confirmed in the article. In order to ensure that the polyurethane (PU) sample is pure and usable, we use a FTIR analyzer. These results confirm that the sample is free of solvent bonds. Finally, these results from this humble research confirm that the PU nanofibre sample is usable in the field of biomedical materials.
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