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A PROTOTYPE FOR A CONTINUOUS NANOFIBER PRODUCTION METHOD

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ABSTRACT

This study aimed to develop a prototype for a new method of continuous nanoscale fiber production without using high-voltage. Two concentrations of alginate (1% and 2%) polymer solutions were prepared and used to fabricate nanofibers in order to test and analyze the performance of the prototype that was designed and built in this study. The production of the fibers was initially tested by obtaining optical microscope images. Scanning electron microscopy (SEM) was used to analyze both the surface morphology and diameter of the selected finest continuous fibers. The SEM images and diameter measurement results confirmed that continuous nanofibers was successfully produced with the prototype developed in this study.

1. INTRODUCTION

Nanomaterials, having better mechanical, optical, magnetic and electrical characteristics than materials at both the micro and macro scales have become a topic of interest in the scientific community [1]. Nanomaterials can be classified based on their dimensionality: particles (0-dimensional), nanorods and nanotubes (1-dimensional), nanofilms (2-dimensional), and more complex nanostructures (3-dimensional) [2]. Nanomaterials are being investigated and used in several different fields including medicine and space due to the advantages nanomaterials can provide. Thus, there is an ever-increasing amount of work involving the use of nanoscale materials in new materials, devices, and systems.

Nanomaterial production techniques are generally classified into two main categories: bottom-up and top-down methods [3-5]. This classification is based on whether the production process begins at the atomic/molecular scale (bottom-up) or from bulk materials (top-down). The primary approaches associated with both bottom-up and top-down techniques are illustrated in Figure 1.

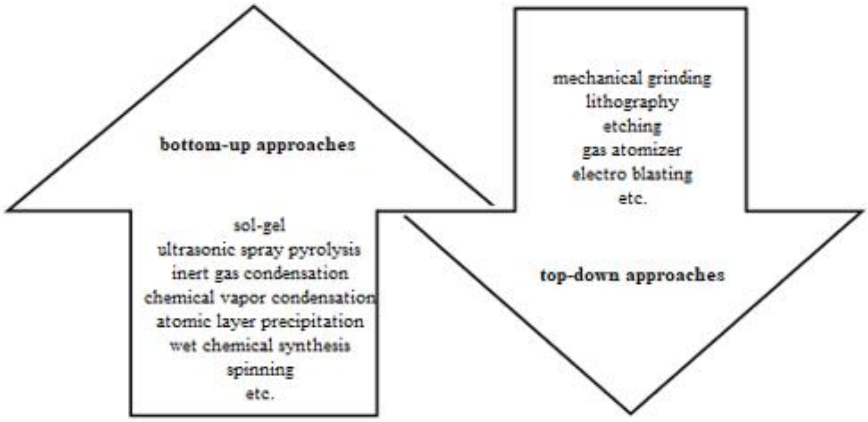

Figure 1. Down-up nanomaterial production (left) and up-down (rigth) approaches [3-5]

Nanofibers are one-dimensional nanomaterials characterized by diameters within the nanoscale range. A variety of techniques have been developed for their fabrication, including phase separation, drawing, template synthesis, self-assembly, centrifugal spinning, and electrospinning [6-8]. Each method offers specific advantages and limitations, which influence their suitability for different applications and contribute to the prevalence of certain techniques over others. Table 1 provides an overview of commonly used nanofiber fabrication methods, key process parameters, and their respective benefits and constraints.

Table 1. Nanofiber fabrication methods, important process parameters, their advantages and limitations [8-11].

Method	**Voltage need**	**Important parameters**	**Advantages**	**Limitations**
Phase Separation	-	Solvent type, polymer concentration and temperature	- Controllable pore size and morphology, - Suitable for biomedical applications	- Only certain polymers can be used. - Not suitable for continuous fiber production. - Limited scalability and time-consuming
Drawing	-	Solution viscosity, Solvent evaporation rate, Pull-away speed, Distance of pulling Plate area (or geometry) Time of stretching Solvent properties	- Very simple, low-cost setup - No high voltage needed. - Good control over fiber alignment and orientation. - High molecular alignment.	- Limited to certain polymers and laboratory production. - Not suitable for continuous fiber production. - Time-consuming.
Template	-	Template pore shape and size	- Controllable fiber diameter, length and shape. - Reproducible. - Highly ordered fiber alignment.	- Need of template, - Limited scalability, - Limited fiber length / less continuous filaments, - Fiber geometry limited to the template
Self-Assembly	-	Building block concentratrion, pH, temperature, ionic strength, interaction strength, time (for self-assembly to happen)	- Extremely fine, often molecular-scale fibers.	- Only certain polymers can be used. - Limited scalability and complex processes. - Very low production rate (not high-throughput) - Weak mechanical strength - Hard to scale for industrial production
Electrospinning	High	Voltage. Solution viscosity, Solvent used. Tip to collector distance.	- Scalable. - Extremely fine fibers. - Large surface area.	- High voltage need. - Solvent evaporation is required. - Environmental sensitivity
Centrifugal spinning		Spinneret rotation speed. Polymer solution / melt viscosity. Hole size of spinneret. Polymer feed rate. Temperature if melt spinning. Distance to collector	- Scalable. - Very high production rate. - No need for very high voltage. - Lower cost per fiber mass.	- Less precise control over fiber diameter than electrospinning - Material limitations (viscosity, surface tension) - Possible instabilities in jet - For some setups, solvent evaporation or cooling can be a challenge

Among the principal reasons why some of the nanofiber fabrication techniques listed in Table 1 remain less prominent are their limited scalability and the inherently slow and constrained nature of their production processes. In contrast, electrospinning has emerged as a leading scalable technique primarily due to its capacity to generate ultrafine fibers with high controllability, uniformity, and reproducibility.

Over time, the electrospinning parameters for producing nanofibers from different polymers and biopolymers have been extensively investigated for various applications [12-17]. In a study, nanofibrous surfaces were fabricated via the electrospinning technique using solutions of varying concentrations, prepared by dissolving polyacrylonitrile (PAN) in dimethylformamide (DMF) and polyvinyl alcohol (PVA) in water. The influence of solution concentration on the resulting nanofiber diameter was systematically investigated. It was reported that the average diameter of a fibre increased in proportion to the amount of polymer in the solution [12]. In another study conducted in 2025, two-layered biocomposites were produced by electrospinning PVA solutions containing different concentrations of Aloe Vera Gel (AVG) onto surfaces fabricated from a poly(L-lactic acid) (PLLA) solution using the electrospinning technique. As a result, it was stated that increasing the AVG content in the mixtures enhanced the structural strength of the material. Some research has shown that as the amount of PVA decreases in a PVA/AVG fiber mixture, the resulting fibers will be smaller [13].

In a study by Deepak et al., a nanogenerator based on electrospun nanofibers was created from V_2CT_x MXene produced by hydrothermal synthesis and acid etching, and PVDF. This device generated 124 volts (open circuit voltage) and 2.2 micro amperes (short circuit current) when touched by a single fingertip [14]. A group of researchers termed Poshina et al. conducted studies on electrospun nanofibers created for tissue engineering applications using methacrylated alginate nanofiber mats fabricated via the electrospinning process. It was stated that it provides a suitable environment for the growth of the selected cells [15]. Temiz and Yurtseven produced composite nanofibers containing TiO_2-PVP-Avocado seed extract (T/P/A) at different polymer concentrations and under various electrospinning parameters, and additionally subjected them to heat treatment at 500°C for 3 hours. Among the productions, it was reported that the use of 5 w% PVP at 15 kV and a 15 cm distance resulted in the formation of uniform nanofibers [16]. Pahalı conducted a study on energy harvesting using a piezoelectric nanogenerator containing polyvinylidene fluoride (PVDF), lead zirconium titanate (PZT), and graphene nanoplatelets (GNP), produced via the electrospinning technique. As a result, it was stated that the fabricated piezoelectric nanogenerators could be suitable for self-powered wearable motion sensors [17].

For successful nanofiber formation, a sufficient quantity of polymer solution must be available; however, polymers with high viscosity have less mobility than lower viscosity polymers, which affects how rapidly they can travel through an electric field. Other key chemical properties that affect how fibers are formed from the solution include the surface tension of the solution and the charge density of the polymer solution [9, 18]. Variability in the distance between the syringe needle's end and the surface of the collector may influence how the fibers are created, and thus they can control how the fibers appear. Increasing this distance or reducing the electric field leads to lower droplet density. Furthermore, If there are charged regions on the collector, the characteristics of the charge in those regions can alter the fiber appearance or change the surface characteristics of the collector. Current research on electrospinning and electrospraying indicates that the morphology and structure of the resulting nanofibers are governed by the synergistic interplay between electrostatic forces and solution properties.

The widespread adoption of electrofiber drawing, known for its ability to continuously produce nanofibers at high speeds, has limited research into alternative fiber production techniques. However, the requirement for high-voltage applications, in the kilovolt range, and the rising costs of related equipment highlight the need for alternative methods for continuous nanofiber production [19]. As an alternative to electrospinning, Weise et al. [19] aimed to produce fine fibers without using an electric field by employing laboratory-scale melt spinning and wet spinning systems. In their study, graphene-doped PVA nanocomposite fibers were produced; however, the fibers obtained were on the micron scale.

In this study, a prototype based on the wet spinning technique was developed to enable the continuous production of nanoscale fibers without the use of high voltage. Using the developed prototype, nanofiber production was successfully achieved, representing a significant advancement and offering a valuable

contribution to the literature due to its potential as an alternative nanofiber production process to electrospinning.

2. MATERIALS AND METHOD

2.1. Materials

The prototype was assembled using a range of components, including a manometer (0–2.5 bar), flowmeter, Arduino microcontroller, 12 V DC motors (125 rpm), power supply, collector shafts, bearings, chipboard, hoses, M12 bolts, a cylindrical container, gaskets, cannulas, clamps, a hot air gun, and a motor speed controller. The components were purchased from local suppliers. ALG polymer, with a density of 1.6 g/cm³, was sourced from Carlo Erba Reagents S.A.S. The viscosities of the prepared polymer solutions were measured using a JKI-JK-RV-1 model viscometer. An optical microscope was used to image the microstructure of the fibers. The scanning electron microscope (SEM) was then used to image selected fibers having the smallest diameter.

2.2. Prototype Setup

A feeding system was utilized to provide a constant supply of polymer solution to create fibers. The fiber formation chamber was developed as a partially enclosed chamber with three sides enclosed to reduce the effect of ambient air currents. The fourth side was left open but could have used plexiglass if required, but ultimately it was not necessary to seal off the fourth side with plexiglass. A proposed design schematic is provided in Figure 2.

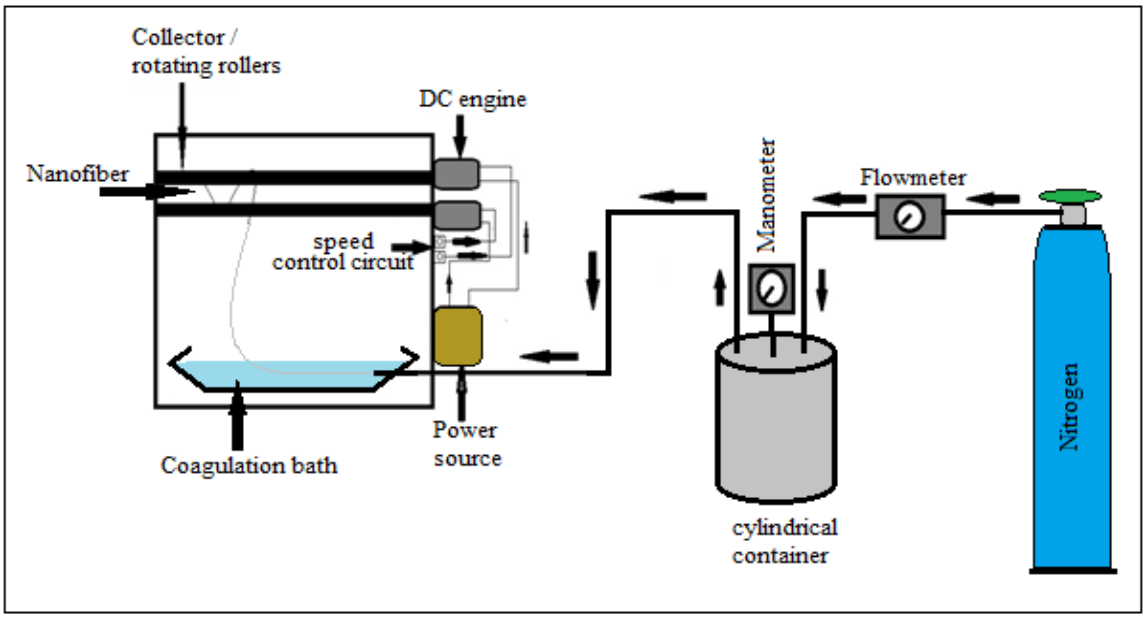

Figure 2. Design of the prototype and workflow.

The foundation of the system was formed by using chipboard sheets with dimensions of: 40 x 50 cm, 40 x 80 cm, 25 x 25 cm, 25 x 35 cm, 50 x 80 cm. The collector shafts have been affixed between the fixed bearings and attached to the 12V motor. To power the collector shafts and motor, these two components were connected to the power supply via wires. Finally, a motor speed control system was used to control the speed of the DC motor up to 125 revolutions per minute.

A carrier system was designed to transport the polymer solution to the cannula. A cylindrical metal container was used for this purpose. Mounting holes were drilled in the container to accommodate hose and manometer connections, and the container was sealed using a gasket. Nitrogen gas was pressurized into the container to drive the polymer solution through the hose. A flowmeter was subsequently connected to control the gas flow, allowing precise regulation of the solution's flow rate. Two additional mounting holes were drilled into the sides of the container for bolt installation. The finalized version of the system is illustrated in Figure 3.

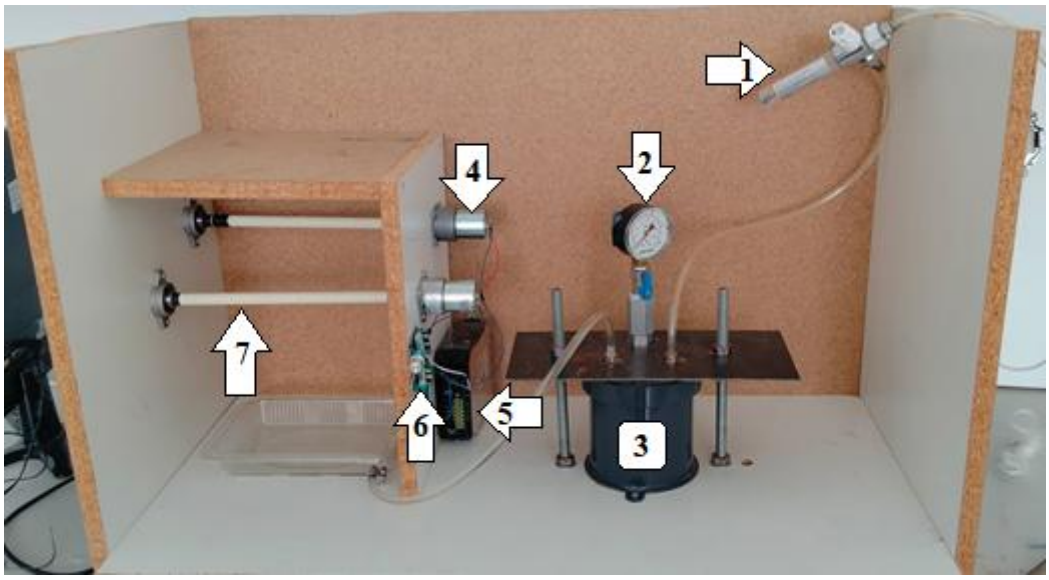

Figure 3. Image of the prototype created for nano-fine fiber production: (1) Flowmeter; (2) Manometer; (3) Cylindrical Metal Container; (4) DC Motor; (5) Power Supply; (6) Motor Speed Control Circuit; (7) Collector Miller

The tools and equipment used in the nano-fine fiber production prototype and their properties are given in Table 2.

Table 2. Tools and materials used in the nano-fine fiber production prototype and their properties

Tool or material	**Properties**	**Purpose of use**
Flowmeter	0-10 liters/min gas flow adjustable	Controlling the amount of gas fed into the system and controlling the amount of solution coming out of the cannula
Manometer	Indicates 0-2.5 bar pressure	Determine the pressure in the tank
Cylindrical Metal Container	It can be filled with up to 1 liter volume and includes a gasket to prevent gas leaks.	Tank to push the solution with gas pressure and move it from the cannula
DC Motor	12 V, 125 rpm	Stretching and winding the fiber obtained from the coagulation bath onto the collector shafts
Power Supply	Amperage can be adjusted between 0-5 A, Voltage can be adjusted between 0-30 V.	Adjusting the voltage and amperage required to run 2 12-volt DC motors
Motor Speed Control Circuit	It can provide speed control of motors in the 6-28 V range.	Controlling the rotation rate of DC motors and aluminum shaft per minute and stretching and winding the fibers on the collector shaft
Collector Miller	Aluminum 1 cm diameter and 30 cm long, 2 pieces	One of them was used to stretch the fiber and the other was used to wind it on the collector.
Bearings	Compatible with 1 cm diameter aluminum shafts.	It can enable the collector shafts to rotate independently of the system.
Hose pipe	1 cm diameter	Used to transfer the gas into the tank and to connect the polymer solution to the cannula.
Cannula	0.6 mm, 0.9 mm, 1.1 mm, and 1.3 mm diameters	Obtaining polymer fibers with different diameters by using cannulas with different diameters

2.3. Preparation of Polymer Solution

ALG is one of the most commonly used polymers due to its high solubility in water. To prepare 1 wt% and 2 wt% ALG solutions, the appropriate amount of ALG polymer was added to distilled water and stirred. The solutions were mixed on a magnetic stirrer at 100 rpm for 3 hours at room temperature to achieve complete polymer dissolution. Alginate solutions were prepared using an IKA® RH basic 2 magnetic stirrer. The visuals illustrating the preparation of alginate polymer solutions are shown in Figure 4.

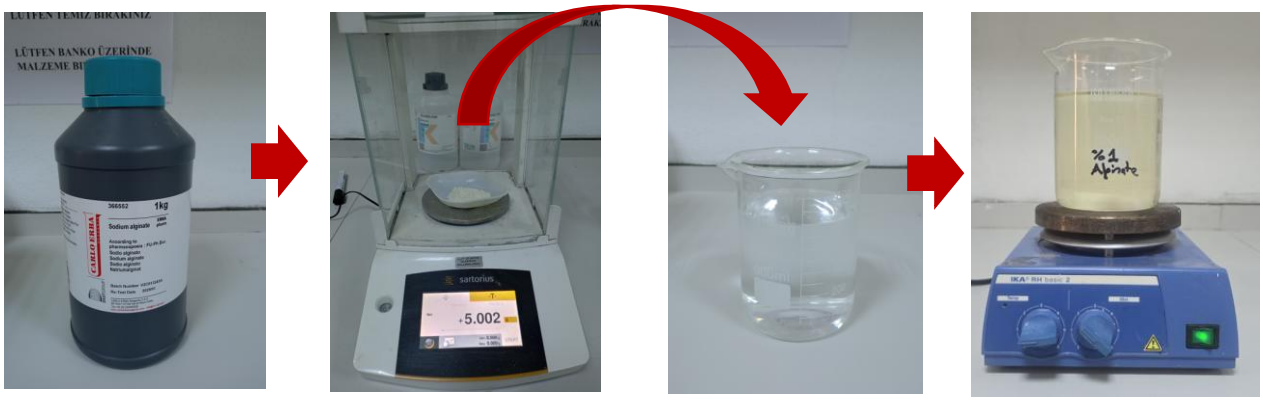

Figure 4. Images of the preparation of alginate solutions.

2.4. Fiber Production

The prepared polymer solution was poured into the cylindrical barrel of the system. To prevent gas leakage during pressurization, the top of the barrel was sealed using a vise-like clamping mechanism with a metal plate. The polymer solution was delivered from the feeding barrel through a hose to a cannula. Several different sizes of cannula were used in this process with diameters measuring 0.6 mm, 0.9 mm, 1.1 mm and 1.3 mm. Each size of cannula was clamped onto the hose to prevent leakage. Upon completion of the assembly of all the components, gas flow was introduced into the cylindrical container. Images were obtained during the fiber creation and can be seen in Figure 5.

Polymer solutions containing 1% and 2% alginate (ALG) were prepared using distilled water. Polymer solutions were stirred on a magnetic stirrer at 100 rpm for 3 hours at room temperature. These solutions were placed in a cylindrical feeder prior to starting gas flow and applying pressure at which point both solutions were able to be extruded from the cannula tip with 5 bar of pressure for the 1% ALG and 10 bars of pressure for the 2% ALG.

Figure 5. Images of fiber production.

The polymer was solidified rapidly using a coagulation bath of 1% calcium chloride ($CaCl_2$) in water to create polymer fibers. When the polymer solution was immersed into the coagulation bath, it began to solidify and form fiber. The rotating collector collected fibers that were formed from the cannula. The speed of the collector was controlled by a DC motor and motor speed controller. Fiber production continued until the barrel containing the polymer solution was empty.

2.5. Optical Microscope and Scanning Electron Microscope Images of Produced Fiber

Initially, the prototype's fibers were evaluated optically to measure fiber diameter and surface characteristics using a SOIF XJP-6A optical microscope. A photograph of the operation of the optical microscope is shown in Figure 6.

Figure 6. Image of the SOIF XJP-6A optical microscope during fiber imaging.

Fiber diameter comparisons were made based on the optical images of the fibers. To obtain more precise visualizations on the surface morphology of the finest fibers, samples from each solution (1% and 2% ALG) were then processed by the JEOL JSM-7100F field emission scanning electron (FE-SEM) and due to being non-conductive the samples were coated with an Au-Pd layer.

3. RESULTS AND DISCUSSION

3.1. Viscosity Measurements of Solutions

Two types of alginate (ALG) solutions were created prior to the fiber-making process and tested using a JKI-JK-RV-1 viscometer to evaluate their viscosities. The unique molecular structure of alginate allows it to quickly gel, which can be attributed to the physical properties of the alginate polymer. Increasing the polymer concentration from 1% to 2% led to a significant increase in viscosity. The measurements showed that the viscosity of the 1% ALG solution was 325 cP, whereas that of the 2% ALG solution was 1262 cP. This significant difference in viscosity was expected to affect the diameters of the produced fibers.

3.2. Fiber Formation and Fiber Diameter Determination under Optical Microscopy

The prototype developed in this study has the ability to produce continuous fibers and that polymer fibers of various thicknesses can be obtained using cannulas of different diameters. As can be seen from the Figure 7, the fibers produced with cannulas of 0.6 mm, 0.9 mm, 1.1 mm, and 1.3 mm diameters have significantly different thicknesses depending on the concentration of the alginate solution used (1% and 2%).

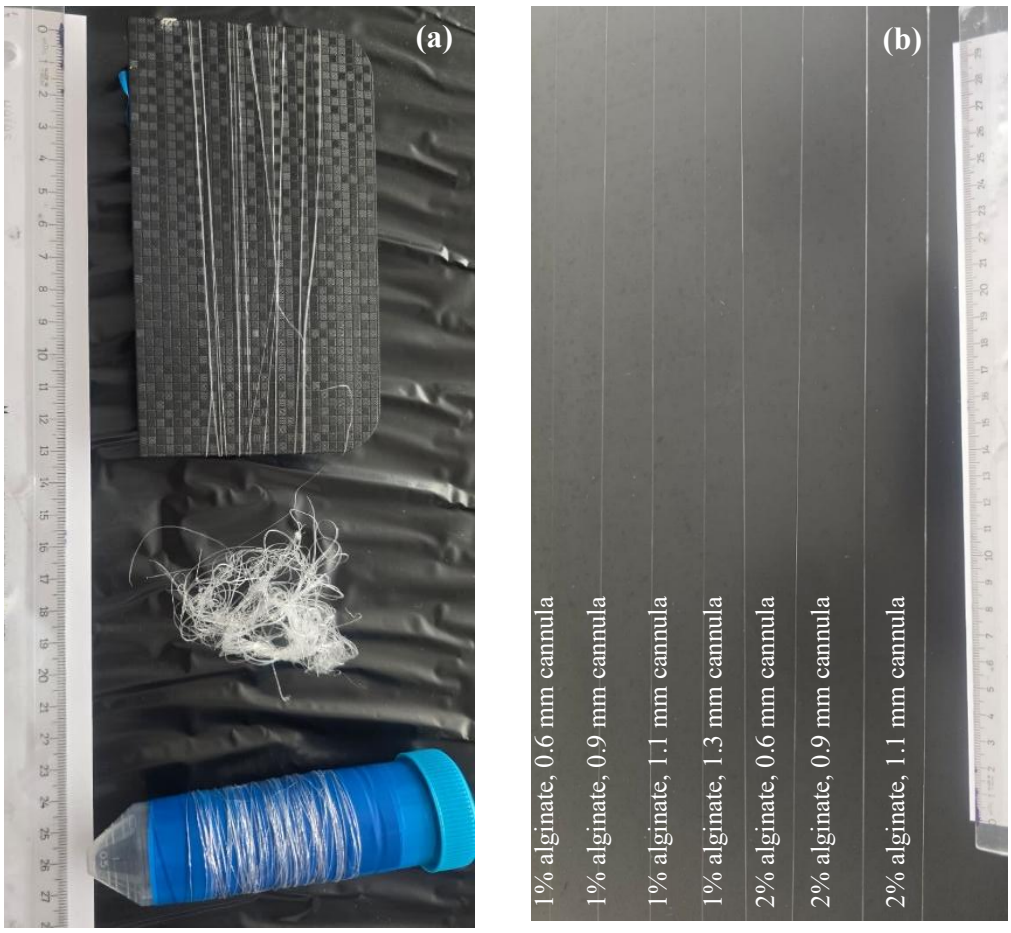

Figure 7. a*) Image showing that continuous fibers can be produced with the prototype. b) Image of polymer fibers with different thicknesses obtained from cannulas with diameters of 0.6 mm, 0.9 mm, 1.1 mm and 1.3 mm and 1 percent and 2 percent alginate solution.*

Optical microscope images of the fibers produced using the prototype developed in this study are presented in Figures 8 and 9. Examination of these images provided preliminary information on fiber diameters and surface morphology. As observed in the optical microscope images presented in Figures 8 and 9, increasing the cannula diameter resulted an incerase in the amount of solution contacting the coagulation bath per unit time therefore an incerase in fiber diameters. Considering the production performed with the 1% ALG solution shown in Figure 8, fibers produced using a 0.6 mm diameter cannula had diameters of approximately 700 nm, whereas production with cannulas of 0.9 mm, 1.1 mm, and 1.3 mm diameters yielded fiber diameters of approximately 6 μm, 8.5 μm, and greater than 10 μm, respectively. Similarly, for the production carried out with the 2% ALG solution shown in Figure 9, fibers produced with 0.6 mm, 0.9 mm, 1.1 mm, and 1.3 mm diameter cannulas exhibited diameters of approximately 2.7 μm, 10.3 μm, 19–20 μm, and greater than 35 μm, respectively.

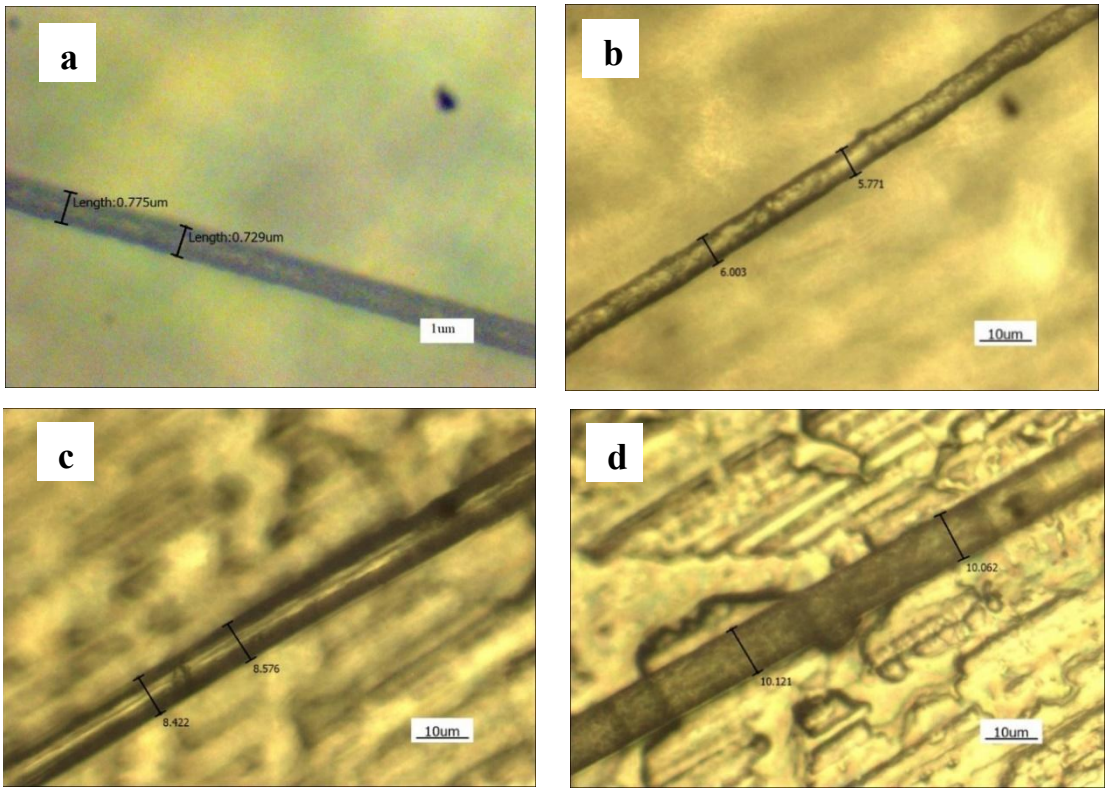

Figure 8. Optical microscope images of 1% ALG fibers produced from different cannula diameters; a*) 0.6 mm; b) 0.9 mm; c) 1.1 mm; d) 1.3 mm*

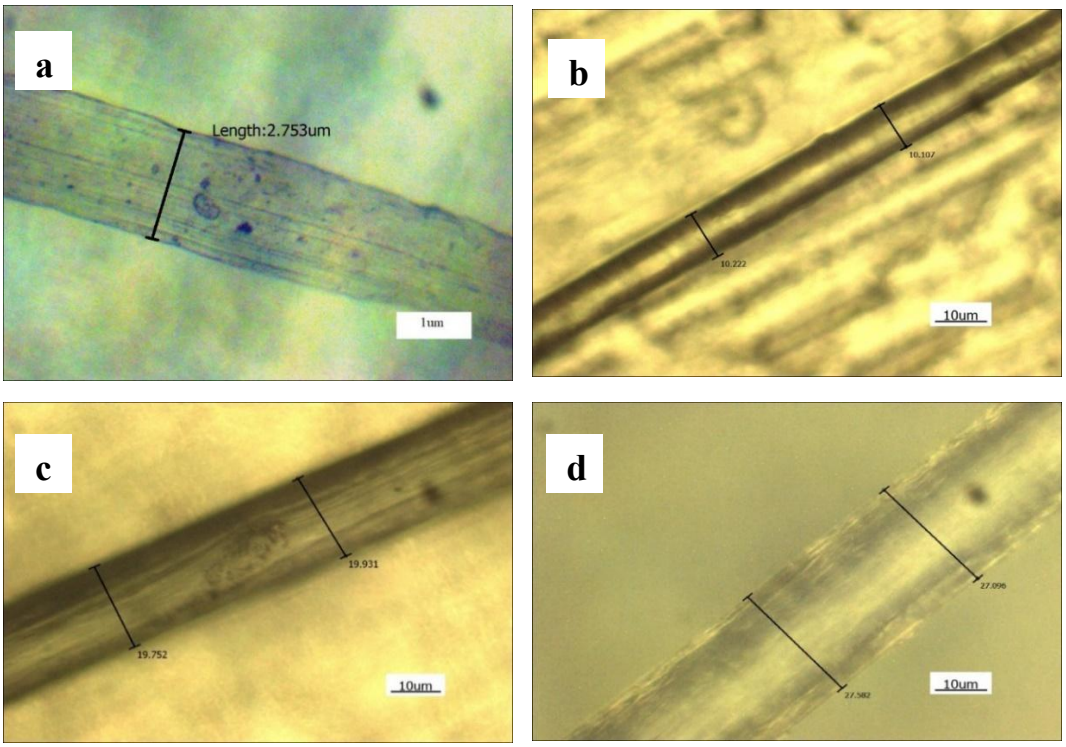

Figure 9. Optical microscope images of 2% ALG fibers produced from different cannula diameters; a*) 0.6 mm; b) 0.9 mm; c) 1.1 mm; d) 1.3 mm*

When the cannula diameter was kept constant but solutions with different ALG concentrations were used, the fiber diameter increased with increasing ALG concentration due to the corresponding rise in

viscosity, which is consistent with literature reports [12–13]. Similarly, when all other parameters were held constant, an increase in cannula diameter also resulted in larger fiber diameters. Fibers produced from the 1% ALG solution by using a 0.6 mm cannula exhibited diameters of less than 1 μm, whereas the fiber diameters of those obtained from the 2% ALG solution ranged between 2 μm and 3 μm. The relationship observed between polymer concentration and fiber diameter is consistent with previous literature reports [20–21].

3.3. Determination of Fiber Morphology under Scanning Electron Microscopy

The fiber formation and diameters, from a few microns to nano-size, were confirmed with optical microscope images. Since the thinnest fibers for both 1% and 2% polymer solutions were obtained using a 0.6 mm diameter cannula, these fibers were used for further morphological analysis. A field emission scanning electron microscopy (FE-SEM) was used to investigate the detailed morphology of the fibers. A thin layer of gold-palladium (Au-Pd) layer was coated onto produced fibers to enhance conductivity and optimize the image quality. After this prep, the samples were mounted to the aluminum stubs, and viewed on the FE-SEM images were taken at high enough voltage to allow resolution of the fiber surface morphology. The SEM images of the selected fibers are presented in Figure 10.

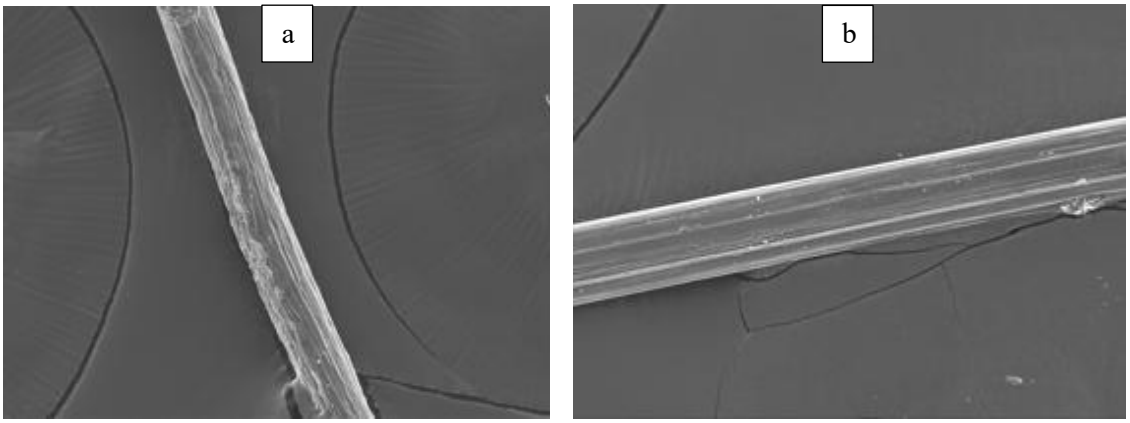

Figure 10. Microscope images of the produced fibers; a*) 1% ALG fiber; b) 2% ALG fiber*

Figure 10 contains images of the nanofibers produced. The fibers have relatively uniform morphologies and are free from large agglomerations and imperfections such as locally thinned and thickened areas. The fibers also have relatively smooth and continuous surfaces, further demonstrating that the fiber production process is stable and reproducible. The similarity of fiber diameter on a macroscopic scale is not sufficient to show equivalency; the diameter of fibers with similar diameters will vary (and therefore be distinguishable) even with the use of optical microscopy and scanning electron microscopy. The results of both imaging modalities confirm that fiber diameter will increase with an increase in ALG concentration in solution and with an increase in cannula diameter. Based on these results, the prototype nanofiber production system has great potential to provide a means of producing continuous nanofibers

4. CONCLUSION

In this research, prototype of a novel nanofiber production system was designed, built and tested. The most important distinction between this new system and electrospinning (the traditional method for producing nanofibers) is that the fiber production system does not require the use of high-voltage electric fields when producing nanofibers. Rather than using an electric field, fibers produced by the fiber production system are made from polymer solution fed under pressure through the fiber-forming device. Fiber diameters were optimized from fiber sizes smaller than 1 micron to as large as 30 microns at the completion of the previous optimization phase using optical microscopy. After optimizing the fiber production system, optical microscopy results indicated that submicron-sized fibers (~700 nm) can be produced using 1% ALG solution with a 0.6-mm cannula, which demonstrates that the fiber production system can produce nanofibers without an applied electric field. The results obtained from this study indicate that the prototype system is capable of being used for producing nanofibers at the nanoscale.

Conversely, the fibers spun with different sized cannulas using 1% ALG solutions were approx. 50% thinner than those using 2% solutions. This suggests that the diameter of the fibers could be reduced even further by applying an increased level of tension to the fibers through the use of differing speed settings between the spinning rollers. Therefore, the focus of future studies will be to develop finer fiber though an accurate means of controlling both the roller speed and feeding mechanism adjustments.

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Conflict of Interest Statement

There is no conflict of interest between the authors.

Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

Artificial Intelligence (AI) Contribution Statement

This manuscript was entirely written, edited, analyzed, and prepared without the assistance of any artificial intelligence (AI) tools. All content, including text, data analysis, and figures, was solely generated by the authors.

Contributions of the Authors

Güray ERKUT: Investigation, visualization, data analysis, writing- original draft preparation.

Ercan ŞENER: Investigation, visualization.

Derman VATANSEVER BAYRAMOL: Conceptualization, methodology, writing- original draft preparation, reviewing and editing, funding acquisition, supervision.

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