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Electrospinsters Final Project Report

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Final Project Report

Electrospinsters

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April 25, 2023

Executive Summary

An electrospinning machine (EM) produces fibers and particles by means of applying a voltage process (electrohydrodynamic phenomena) to a polymer solution by incorporating the use of a receptacle, a pump, a high voltage power supply (HVPS) and a collector.

EMs are typically very expensive, however, there has been work conducted by various researchers to construct in-house machines at a much lower cost. The growing applications for electrospinning continue to be a source of interest for many researchers as it is still a relatively new process. Much of the effort has been dedicated to producing nanofibers with unique properties with a focus on improving the efficiency and scalability of the process.

The Electrospinsters Senior Design Team are researching and designing an in-house EM that can produce nanofibers for the team sponsor's research and serve other educational purposes at Trinity University. The sponsor, Dr. Dany Munoz-Pinto, intends to use the results of this project to expand his research projects and goals by incorporating nanofibers into tissue scaffolds. The prototype must be a functioning EM so that a future team or the sponsor's research students can make additions, but not struggle with the basic functions to create nanofibers.

Based on published literature and additional research conducted by the team, we determined that an EM is composed of four subsystems: a syringe pump, a HVPS, a collector, and a user interface. The HVPS provides a voltage to the solution in the syringe pump which when exuded is drawn to the grounded collector due to the difference in electric potential. This drawn-out solution conglomerates on the collector which forms the scaffold. Published literature allowed us to gain a better understanding of the setup and we learned that there is not much variation in how the EM can be modified. Consequently, we chose to follow a fundamental setup with a flat collector plate due to its easy construction and compatibility with producing non-woven nanofibers with polyvinyl alcohol (PVA).

We designed and conducted a series of tests to validate the subsystems of the device and to test the EM against various design constraints and project requirements. Some of our constraints pertained to time and budget and our project met both of these, as we successfully created a working prototype for our sponsor by the end of the 2023 spring semester, and we only used \$808.92 of our \$1200 budget. Other criteria related to health and safety were met, since we complied with TU Environmental Health and Safety and OSHA standards, the voltage applied to the solution did not exceed 30 kV at any point during testing and application, and our device fit dimensional constraints and was only operated in a CSI fume hood to prevent the inhalation of nanoparticles. Our prototype operates all electrical subsystems using US standard outlets. Certain requirements correlated with certain subsystems which had specific tests designed to evaluate the flow rate, voltage, voltage display, and nanofiber diameter. The Flow Rate Variability Test evaluated the syringe pump subsystem with variable flow rates of 0.5 mL/hr, 1.0 mL/hr, and 1.5 mL/hr and deemed accurate enough for testing purposes. The Voltage Variability Test tested the active voltage of the HVPS and verified its operation is within a $\pm 5\%$ margin of error. The Proof-of-Concept Test verified that the EM could produce non-woven nanofibers of 200 nm and that it is within $\pm 20\%$ error of previously published experiments, which are acceptable results for our sponsor's research purposes. Additionally, we tested Tip Diameter Variability and Collection Distance Variability to observe the effects on the nanofiber diameter and determined that there is not a significant difference as they are still within $\pm 20\%$ error, as we had expected from published literature.

Overall, the Electrospinsters created a successful, working prototype to aid in our sponsor's research. Our prototype met all requirements and constraints, and there are no remaining changes needed to achieve our final goals. However, for further improvements, we hope that a future team will improve this final prototype by integrating another type of collector that can produce aligned nanofibers while maintaining the ability to interchange collector types and implementing any other useful additions or modifications.

Introduction

The problem this project deals with is the time-consuming and difficult process for creating scaffolding in tissue culture, which is important for the structured growth of cells. Electrospinning offers a quick and efficient method for creating polymer-based nanofiber webbing that works well as scaffolding. However, the high cost of electrospinning machines is a barrier for researchers, including our project sponsor. The goal of this project is to construct an affordable electrospinning machine that will benefit our sponsor's research experiments by making the scaffolding process require less human interaction. The project aims to design, model, implement and test an Electrospinning Machine (EM) to be used for research and educational purposes by Trinity University. The schedule objectives were to have prototype testing done by March 10, 2023, and project closeout and delivery of a final prototype by April 28, 2023. The device must run off a 120V 60Hz AC outlet, and not exceed dimensions of 1.5 ft x 3 ft x 2 ft. The voltage applied to the solution should not exceed 30 kV at any point during testing, and a display shall always show the active voltage of the high voltage power supply (HVPS), within a $\pm 20\%$ margin of error. The syringe pump should function with variable flow rates of 0.5 – 1.5 mL/hr based on user input. The dimensions of the nanofiber should be within $\pm 20\%$ error of previously published experiments, and the proof of concept shall use polyvinyl alcohol (PVA) solution to produce non-woven nanofibers of 200 nm $\pm 20\%$. To meet these objectives, we purchased an HVPS with the capability to reach up to 40 kV. We added a multimeter in combination with a voltage divider to our original design to show one thousandth of the voltage being applied to the solution during the process. The syringe pump purchase was also tested within the desired ranges to ensure proper functionality. Finally, nanofibers were characterized using a scanning electron microscope (SEM) to determine fiber diameters.

Overview of the Final Design

Subsystem Designs

This section identifies the features of the electrospinning machine that we tested against our working criteria and identifies the main subsystems that were tested separately before assembling the complete prototype. The four main subsystems are the syringe pump, collection plate, high voltage power supply, and the voltage safety display. All subsystems required individual testing.

Syringe Pump

The syringe pump subsystem serves to exude the polymer solution that enables nanofiber production. Since syringe pump designs do not vary significantly, and it is part of a niche market, there were limited options to consider into our design process for this subsystem. Taking this into account we decided to purchase an NE-300 Just Infusion™ syringe pump, as seen in Figure 1 from a verified distributor of the New Era Instruments brand. We looked at the user's manual for syringe pump for guidance on how to appropriately set up the pump. For the success of the EM, the syringe pump must have accurate and precise flow rates. To test this, we created a flow rate variability test. In performing this test, we wanted to ensure basic functionality of the pump as an independent subsystem.



Figure 1. NE-300 Just Infusion Syringe Pump

High Voltage Power Supply

The HVPS subsystem creates an electric potential between the polymer solution and the collection plate, creating the driving force for drawing out the desired fibers. This device was essential to the success of the EM; thus, it was crucial to test the device and ensure it was functioning properly. Similar to the syringe pump subsystem, we noted that precision and accuracy and especially safety were critical to creating the EM. Therefore, the main factors we considered were the output voltage range and the cost of such a device. For these reasons, we decided to purchase the HVPS seen in Figure 2 since it was cost-effective and met the voltage output range needed to produce the non-woven nanofibers. To ensure safety, we followed the guidelines set in the device's safety manual and the guidelines given to us by Trinity's Health and Safety Department. Since the electric potential is a significant factor on fiber success and geometry it was important to be able to produce a variety of voltages with minimal error. To assure this, we performed a voltage test that would test a variety of voltages and ensure the basic function of the device. The device produced variable voltages between 5 kV and 30 kV.



Figure 2. High Voltage Power Supply

Collection Plate

The collection plate subsystem allows us to collect the nanofibers safely for careful assessment and characterization using the SEM. This subsystem involves a simple flat plate collector that serves to collect the non-woven nanofibers and complements the objectives of our project. Flat plate collectors allow for the creation of random/non-woven fibers whereas other options are geared towards aligned orientation of fibers over a large area or the creation of electrospun particles. [1] Therefore, we decided to only test the flat plate collector for this semester with the implementation of a modular design such that future design teams could test other collector types. The flat collector plate required minimal effort to design or manufacture, so we decided to cut and fillet a metal sheet from the Makerspace using a manual metal cutter. Since the use of the collection plate depends on the function of the electrospinning machine, we tested it at variable collection distances. Ideally, the collection plate should collect the nanofibers which can then be characterized using the SEM and proper observations can be made on the collection distance's impact on the production of nanofibers.

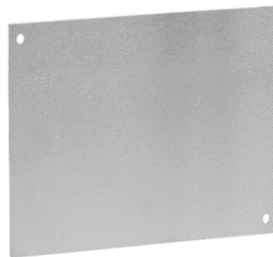


Figure 3. Flat Plate Collector

Voltage Safety Display

The voltage safety display consists of a digital multimeter and a high-voltage voltage divider. The voltage divider consists of a set of resistors, including a special 1 G Ω resistor rated for 50 kV, that provide two summative impedance values that when the multimeter display is wired into the system an output voltage that is one thousandth of the input voltage is produced. A purchased multimeter was decided on, shown in Figure 4, since multiple attempts to code a microprocessor to read the voltage proved to be highly inaccurate and more susceptible to electromagnetic interference. The voltage divider required multiple iterations since our 1 G Ω resistor was not measurable with our available equipment and the resistor we purchased can have a $\pm 20\%$ variance in resistance value. This voltage divider is contained within a plastic 3D printed casing to prevent arcing and keep exposure to high voltage to a minimum. The voltage divider has two long wires that are the connection points for the multimeter to interact with the voltage divider. The length of the wire allows the voltage to be monitored from outside the fume hood while keeping the voltage divider within the fume hood. This is essential for accurate readings since minor fluctuations in high voltages produce electromagnetic waves, much greater than standard voltage fluctuation waves, that are capable of interfering in readings. The metal construction of the fume hood helps contain the waves through absorption and reflection and containing the voltage divider within the fume hood was more practical than constructing a container. The voltage is reduced to one thousandth of the true voltage so the reading on the digital multimeter can be converted to the true voltage simply by treating the V unit on the multimeter as a kV unit. This helps ensure the safety of the user by providing knowledge of a high voltage and indicating to the user the severity of the voltage all from a safe distance. Since this system is essential to providing a safe environment for the user and to achieving the proper voltage, a critical factor in the success of electrospinning, it was crucial to verify this system's validity. This system was compared to another multimeter with an industry standard one thousand to one stepdown probe to verify the accuracy of our system. We knew the probe's readings to be within an acceptably accurate range since the probe has no more than 2% error when paired with the multimeter we used, according to its user manual. The two systems were compared at voltages ranging from 5kV and 30kV in 5kV increments. At each voltage point the two systems were compared five times in order to ensure the consistent success of the system.

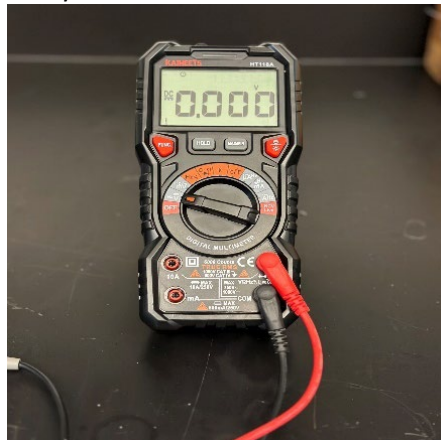


Figure 4. Voltage Safety Display

Design Evaluation

Design Constraints

Constraint 1: The device and all prototypes must be built within the \$1,200 budget given by the department.

Evaluation

The Electrospinsters satisfied this constraint by spending \$808.92 out of the \$1,200 budget.

Constraint 2: The device, being a piece of Trinity University equipment, must meet the safety standards given to us by TU Environmental Health and Safety and OSHA.

Evaluation

The Electrospinsters satisfied this constraint by meeting with TU's Environmental Health and Safety Manager, Jake Hernandez, to minimize risks and ensure we were complying with Health and Safety.

Constraint 3: The device must fit within the university CSI fume hoods to avoid the inhalation of nanoparticles.

Evaluation

The Electrospinsters satisfied this constraint by always operating the EM inside the fume hood.

Constraint 4: The device must be finished by the end of the 2023 spring semester.

Evaluation

The Electrospinsters satisfied this constraint by successfully providing a working prototype of the EM for their sponsor.

Project Requirements

Requirement 1: The proof of concept shall use PVA solution to produce non-woven nanofibers of ~200 nm.

Test: Proof-of-Concept Test

Summary

The Proof-of-Concept Test will serve to evaluate the functionality of the entire machine to produce non-woven nanofibers. This test will reveal how each subsystem interacts with one another, as well as determine how the user will interact with the machine as the process is performed.

Objective(s)

This test is designed to ensure that all the subsystems can operate as a single unit to create the desired non-woven nanofibers of ~200 nm diameter ($\pm 20\%$).

Feature(s) Evaluated

Since this test is inclusive of all subsystems, it will assess the compatibility of the subsystems working together and will assess the order for operating the electrospinning machine. This test will also determine the ease of use of the entire machine.

Scope and Key Test Conditions

The key test conditions are a 1.0 mL/hr flow rate, a 0.51 mm diameter syringe tip, a 12 cm collection distance, and an applied 30 kV voltage difference. Lastly, the PVA solution will be at a 10% w/v concentration.

Assumptions

All our subsystems are working properly, and all other tests (except the tip and collection distance variability) have been successful prior to this test being conducted.

Acceptance Criteria

This test will be considered successful if the entire electrospinning machine can produce non-woven nanofibers of ~ 200 nm ($\pm 20\%$).

Test Results

We conducted four iterations of this test. The first iteration was stopped after 30 minutes due to the power supply from the syringe pump turning off. The second iteration was unable to be completed due to the adapter for the syringe pump failing, due to a buildup of static charges on the syringe pump from the high voltages. The third and fourth tests were the most successful, as we were able to run the electrospinning machine for 5 hours each time and collected the nanofibers seen in Appendix B. After analyzing the samples using the SEM, we determined the nanofibers averaged 228 nm in diameter. Overall, the average percent difference for the different fibers measured was 12.4%. Therefore, the test passed. Figures 5 and 6 below show the nanofibers created using the EM as well as measured nanofibers using the SEM. Further images taken can be seen in Appendix B. The images show a complex, randomly aligned fiber network which is consistent with what we expected to see from using a flat collector plate.

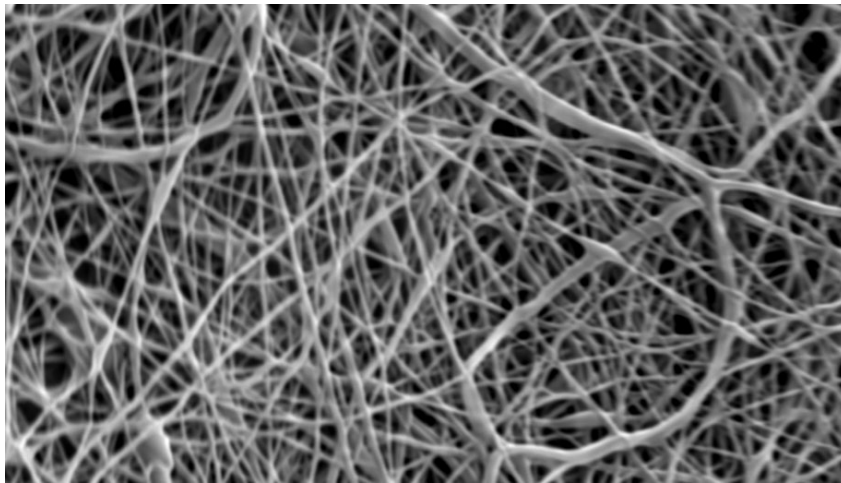


Figure 5. Nanofibers created from proof-of-concept test.

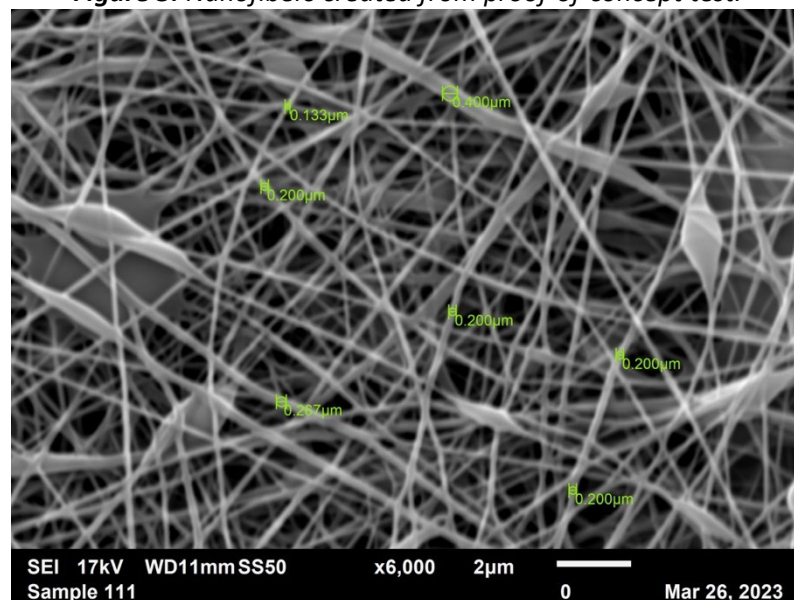


Figure 6. Nanofibers from proof-of-concept test shown with dimensions.

To evaluate the nanofibers on the SEM the thickest section of each PVA mat was cut and coated with 5.5 nm of gold using a sputter coater. The mat was then placed on the SEM and four images per mat were taken for measurements, with 7-10 nanofibers per “section” or image taken at a time. The table below shows the averages and percent differences per section of the mat of nanofibers evaluated. Sections 1-4 were taken from the third test and sections 5-8 were taken from the fourth test.

Table 1. Evaluation of nanofibers from the Proof-of-Concept Test

Section	Average Diameter (nm)	Percent Difference (%)
1	207.4	3.59
2	190.4	5.03
3	214.4	6.73
4	228.6	12.5
5	270.8	26.13
6	259.1	22.81
7	222.3	10.04
8	233.4	14.31

Evaluation

The test met the project requirement and acceptance criteria based on data collected from SEM images.

Requirement 2: The device must run off a 120V 60Hz AC outlet, the standard outlet at Trinity Institutions.

Evaluation

The Electrospinsters met this requirement by operating all our electrical subsystems using US standard outlets.

Requirement 3: The electrospinning machine shall not exceed dimensions of 1.5 ft x 3 ft x 2 ft.

Test: Dimensional Constraints Test

Summary

The Dimensional Constraints Test will assess the physical dimensions of the electrospinning machine.

Objective(s)

This test is designed to confirm that the device fits within the dimensions of the university's fume hoods.

Feature(s) Evaluated

The test will examine the syringe pump, collection plate, high voltage power supply, and voltage safety display, determining whether the single operating unit meets the dimensional constraints.

Scope and Key Test Conditions

This test will consist of simply using the fume hood as storage for the EM.

Assumptions

No specific assumptions were made.

Acceptance Criteria

The test will be considered successful if the machine does not exceed the dimensional constraints of 1.5 ft x 3 ft x 2 ft and fits in our workspace fume hood.

Test Results

The test was considered successful as the measured dimensions of the machine were well within the margins of the required dimensions. The measured dimensions were 1.18 ft x 2.59 ft x 0.38 ft. Therefore, it's safe to say that the machine can fit in any fume hood at Trinity University.

Evaluation

This test was successful and confirmed our prototype adhered to the requirement above.

Requirement 4: The voltage applied to the solution should not exceed 30 kV at any point during testing and application.

Evaluation

This requirement was met as a voltage above 30 kV with a 5% margin was not applied to the PVA solution throughout the electrospinning process.

Requirement 5: A display shall always show the active voltage of the HVPS, within a $\pm 5\%$ margin of error.

Test: Voltage Safety Display Test

Summary

The Voltage Safety Display Test will assess the accuracy of the displayed voltage across a range of voltages.

Objective(s)

This test is designed to ensure that the voltage safety display can evaluate and display the voltage within a $\pm 5\%$ error.

Feature(s) Evaluated

This test will evaluate the voltage safety display and its ability to accurately measure and display voltages within our operable range. This test is essential for the safety and ease of use of this subsystem.

Scope and Key Test Conditions

This test will evaluate the display and will utilize five samples per desired voltage between 5 kV - 30 kV. A digital multimeter will assist in the verification of the true voltage.

Assumptions

The values displayed on the digital multimeter are the true voltage output of the HVPS.

Acceptance Criteria

This test is considered successful if none of the recorded displayed values are different from the true voltage by more than 5% of the true voltage value.

Test Results

The test was considered successful as our final design of the Safety Display never had a percent error exceeding $\pm 5\%$ when compared to the multimeter readings representing the true voltage of the HVPS. This data is contained within Table 2. It is thus safe to say that the Voltage Safety Display is an accurate representation of the true voltage of the HVPS.

Table 2. DMM and Safety Display comparison

Desired Voltage (kV)	Measurement Type	Reading 1	Reading 2	Reading 3	Reading 4	Reading 5
5	DMM Voltage (kV)	5.11	5.11	5.11	5.12	5.11
	Safety Display Voltage (kV)	4.89	4.90	4.90	4.90	4.89
	Percent Error (%)	4.39	4.35	4.37	4.32	4.35
10	DMM Voltage (kV)	9.93	9.93	9.94	9.94	9.95
	Safety Display Voltage (kV)	9.51	9.53	9.53	9.52	9.53
	Percent Error (%)	4.41	4.20	4.32	4.44	4.37
15	DMM Voltage	15.17	15.16	15.18	15.20	15.20
	Safety Display Voltage (kV)	14.75	14.70	14.70	14.69	14.67
	Percent Error (%)	2.86	3.15	3.26	3.48	3.62
20	DMM Voltage (kV)	20.13	20.15	20.16	20.19	20.19
	Safety Display Voltage (kV)	19.87	19.86	20.23	19.84	19.85
	Percent Error (%)	1.29	1.47	0.37	1.74	1.69
25	DMM Voltage (kV)	24.74	24.75	24.74	24.73	24.74
	Safety Display Voltage (kV)	24.36	24.31	24.49	24.42	24.19
	Percent Error (%)	1.56	1.83	1.02	1.26	2.27

30	DMM Voltage (kV)	29.964	29.972	29.984	29.994	29.999
	Safety Display Voltage (kV)	30.75	30.42	30.16	30.24	30.09
	Percent Error (%)	2.56	1.47	0.58	0.81	0.30

Evaluation

The test was successful and showed that our voltage safety display could accurately display the true voltage of the HVPS by meeting the tests requirements.

Requirement 6: The syringe pump should function with variable flow rates of 0.5 – 1.5 mL/hr based on user input.

Test: Flow Rate Variability Test

Summary

The Flow Rate Variability Test will assess the ability of the syringe pump to operate at variable flow rates.

Objective(s)

The test is intended to evaluate the requirement that the syringe pump should function with variable rates of 0.5 – 1.5 mL/hr based on user input.

Feature(s) Evaluated

The Flow Rate Variability test will evaluate the functionality of the syringe pump feature of the prototype, assessing the ability of the device to operate at different flow rates.

Scope and Key Test Conditions

This test will evaluate the syringe pump and the device's ability to operate at a flow rate specified by the user that is within the flow rate range requirement. We will test 3 flow rates, 0.5 mL/hr, 1.0 mL/hr, and 1.5 mL/hr. It will include 5-10 samples of fluid for each flow rate. For each sample, a timer will be set and a collection beaker for the fluid dispensed will be used to calculate the flow rate and compare it to the flow rate set using the syringe pump. The water collected in the beaker will be weighed and divided by the density of water to determine the volume dispensed.

Assumptions

The density of water was 0.997 kg/m³ for all our samples.

Acceptance Criteria

The test will be considered successful if each sample flow rate is within 5% of the ideal flow rate set by the user using the syringe pump.

Test Results

Our initial testing method to determine the reliability of our syringe pump's flow rate was done by setting a tube to collect water over a fixed time and using the tick marks on the tube to measure how much water was collected. Table 3 shows the results we were able to obtain over two samples for each of the respective flow rates presented. While this method produced successful results based on our acceptance criteria, we felt that this was not the most reliable and valid scientific procedure to measure volume. Even though the syringe pump manual does state that the accuracy is within $\pm 1\%$, we also noted that we needed to verify that the set flow rate was precise and accurate to proceed with our other subsystems to put our prototype together. Table 4 displays the results of the new iteration of the flow rate test which suggest the syringe pump is less accurate than expected from our previous method. However, there are multiple factors such

as evaporative water loss and instrumental bias which led to obtaining less than ideal results. Because the amount of liquid being collected was significantly small, it is highly probable that the beaker left open to the atmosphere would result in some water evaporating over time depending on the temperature and humidity in the room. Furthermore, instrumental bias or systematic error of the scale we used to measure the weight of the beaker before and after water was collected could have caused measurements to be consistently lower than the true value of our samples. A combination of factors such as these were most likely causing a higher percent error from the ideal flow rate values, especially at lower flow rates since the mass of the liquid is lower and the effect of evaporation and instrument error is more significant. Based on these results, we are confident that there were external factors affecting our measurements and while there is not much that can be done to reduce the error with this testing methodology, there was a consistent bias associated with water loss and instrumental error in our test.

Table 3. Flow rate test results for initial testing method

Ideal Flow Rate (mL/hr)	Average Exp. Flow Rate (mL/hr)	Average Percent Error (%)
0.5	0.49	2.00
1.0	0.98	2.50
1.5	1.49	0.50

Table 4. Flow rate test results for new testing method

Ideal Flow Rate (mL/hr)	Average Exp. Flow Rate (mL/hr)	Average Percent Error (%)
0.5	0.37	26.19
1.0	0.90	10.01
1.5	1.41	6.21

Table 5 interprets the data and samples collected from the scale weighing method and it indicates a general increase in variation and bias as the flow rate decreases. The coefficient of variation helps determine the variability of each data set for the respective flow rates. A higher coefficient of variation, as seen with the 0.5 mL/hr data, indicates a greater spread and higher degree of variability. The bias calculations determined that the experimentally calculated flow rate was consistently lower with respect to the ideal flow rate for each sample group. This suggests that there is a general combination of instrumental and systematic error that is not unique to one data set.

Table 5. Statistical calculations for new testing method

Ideal Flow Rate (mL/hr)	Coefficient of Variation (%)	Bias (mL/hr)
0.5	8.37	0.13
1.0	1.53	0.10
1.5	2.05	0.09

Another method we could have used to validate this test would have been measuring the volume the syringe had been displaced. This would have consisted of allowing the syringe pump to operate until the syringe had compressed for example, 1 mL for the 0.5 mL/hr test, and similarly adjusting for our other flow rates. Based on the timer set for each flow rate, the flow rate could be easily determined by dividing the amount of volume the syringe had been displaced over the total time it had been operating for. This method would have presented a lower potential for external factors from interfering with our sample collection.

Evaluation

This test satisfied the project requirement based on our statistical analysis of the samples collected even if it did not meet the acceptance criteria.

Requirement 7: The dimensions of the nanofiber should be within $\pm 20\%$ error of previously published experiments.

Test: Proof-of-Concept Test

The test description, results, and evaluation can be found in Project Requirement 1: The proof of concept shall use PVA solution to produce non-woven nanofibers of ~ 200 nm.

Test: Tip Diameter Variability Test

Summary

The Tip Diameter Variability Test will employ a variety of tip sizes to the use of the electrospinning machine to ensure the machine can consistently produce nanofibers of ~ 200 nm diameter.

Objective(s)

This test is designed to evaluate the requirement that the proof-of-concept shall use PVA solution to produce non-woven nanofibers of ~ 200 nm diameter.

Feature(s) Evaluated

This test will assess the syringe pump feature of the prototype, determining its capabilities to produce the same required nanofiber using different tip diameters. This will determine the manufacturability of the fibers.

Scope and Key Test Conditions

The tests will include 10-15 samples of nanofiber collection at a fixed flow rate of 1.0 mL/hr, collection plate distance of 12 cm, and voltage of 30 kV for each tip diameter, 0.51 mm, 0.61 mm, and 0.83 mm and each test was run for 5 hours.

Assumptions

Based on previous literature, we do not expect the tip diameter to affect the diameter of the nanofibers [2].

Acceptance Criteria

If the SEM indicates that all fibers created by the electrospinning machine are within a $\pm 20\%$ error for the 200 nm diameter requirement, the test will have been successful.

Test Results

Figures 7-9 show the nanofibers with corresponding measurements taken using the SEM. Similar to the Proof-of-Concept Test, the fiber alignment shown here is random. Further images taken and used to conduct analysis of the samples can be found in Appendix B.

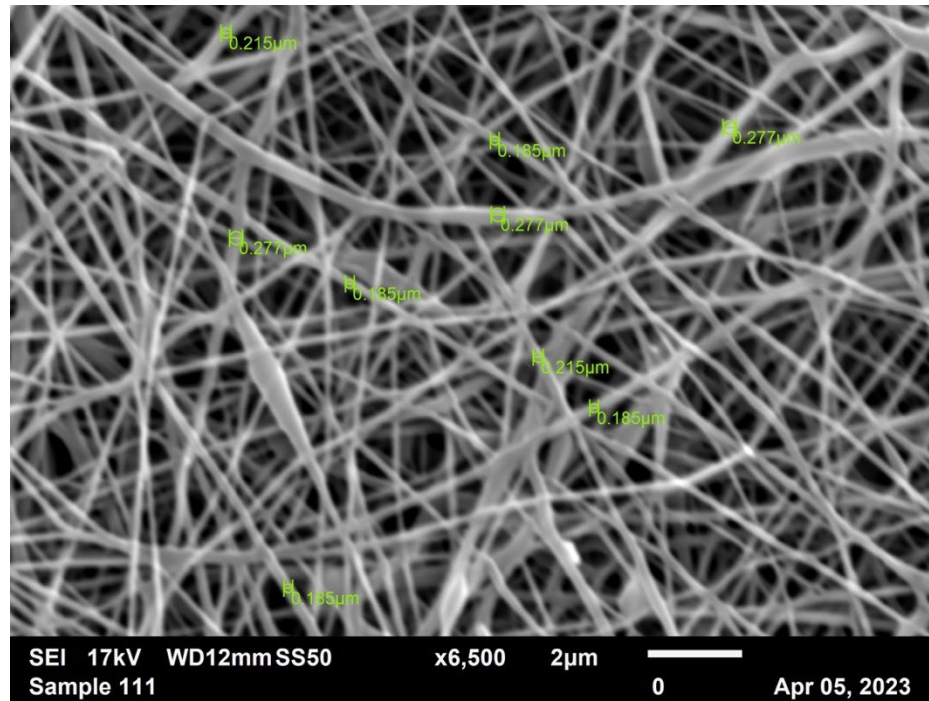


Figure 7. Nanofibers from 0.51 mm tip diameter test with corresponding diameters

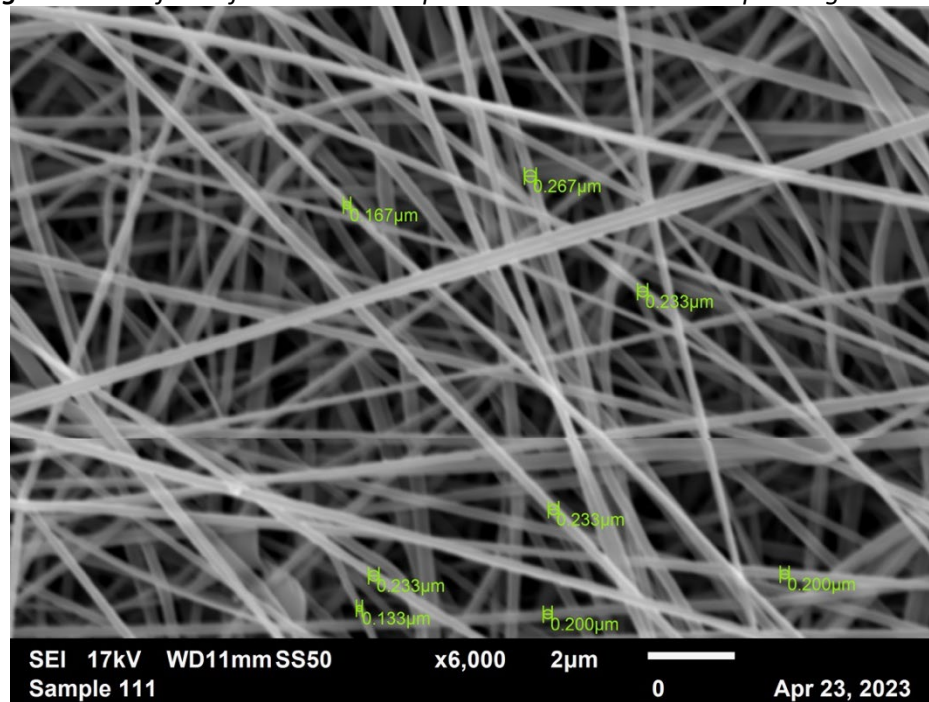


Figure 8. Nanofibers from 0.61 mm tip diameter test with corresponding diameters.

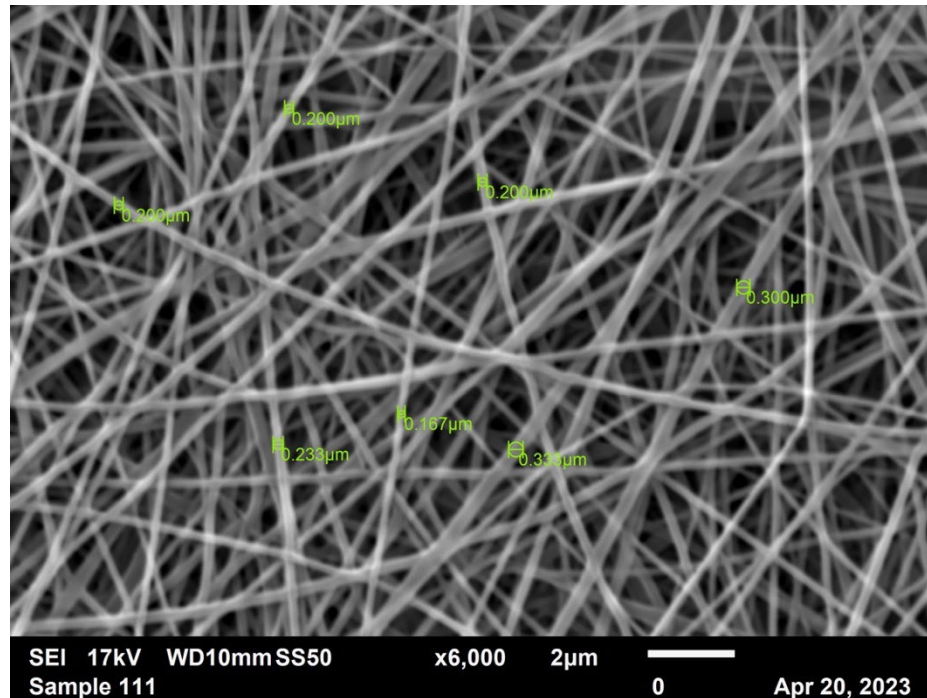


Figure 9. Nanofibers from 0.83 mm tip diameter test with corresponding diameters.

To evaluate the nanofibers on the SEM the thickest section of each PVA mat was cut and coated with 5.5 nm of gold using a sputter coater. The mat was then placed on the SEM and four images per mat were taken for measurements, with 7-10 nanofibers per “section” or image taken at a time. Each test for each tip diameter had four images taken per sample. Table 6 shows the averages and percent differences for each corresponding tip diameter.

Table 6. Evaluation of nanofibers from the Tip Diameter Variability Test

Tip Diameter (mm)	Average Diameter (nm)	Percent Difference (%)
0.51	228.31	12.40
0.61	238.14	16.02
0.83	218.79	8.59

Evaluation

The test met the project requirement as all percent differences for each tip diameter was below 20%. The assumption made that the tip diameter would not have an impact on the diameters of the nanofibers was correct.

Test: Collection Distance Variability

Summary

The Collector Distance Variability test will assess the impact of the collector’s distance on nanofiber production.

Objective(s)

The test is intended to evaluate the objective that the prototype shall use PVA solution to produce non-woven nanofibers of ~ 200 nm diameter.

Feature(s) Evaluated

This test will evaluate the functionality of the collection plate feature of the prototype, assessing the ability to change its distance from the electrospinning machine and how it affects the size of the nanofibers produced.

Scope and Key Test Conditions

The test will evaluate the prototype and utilize 10-15 samples for different collection distances, 10 cm, 12 cm, and 14 cm. The same flow rate of 1.0 mL/hr, tip diameter of 0.51 mm, and applied voltage of 30 kV will be used for all samples taken and the SEM will aid in characterizing the size of the nanofibers.

Assumptions

Based on previous literature, we do not expect the tip diameter to affect the diameter of the nanofibers. [2]

Acceptance Criteria

If the SEM indicated that the fibers created by the electrospinning machine are within a $\pm 20\%$ error for the 200 nm diameter requirement, the test will have been successful.

Test Results

Figures 10-12 show the nanofibers with corresponding measurements taken using the SEM. Similar to the proof-of-concept test, the fiber alignment shown here is random. Further images taken and used to conduct analysis of the samples can be found in Appendix B.

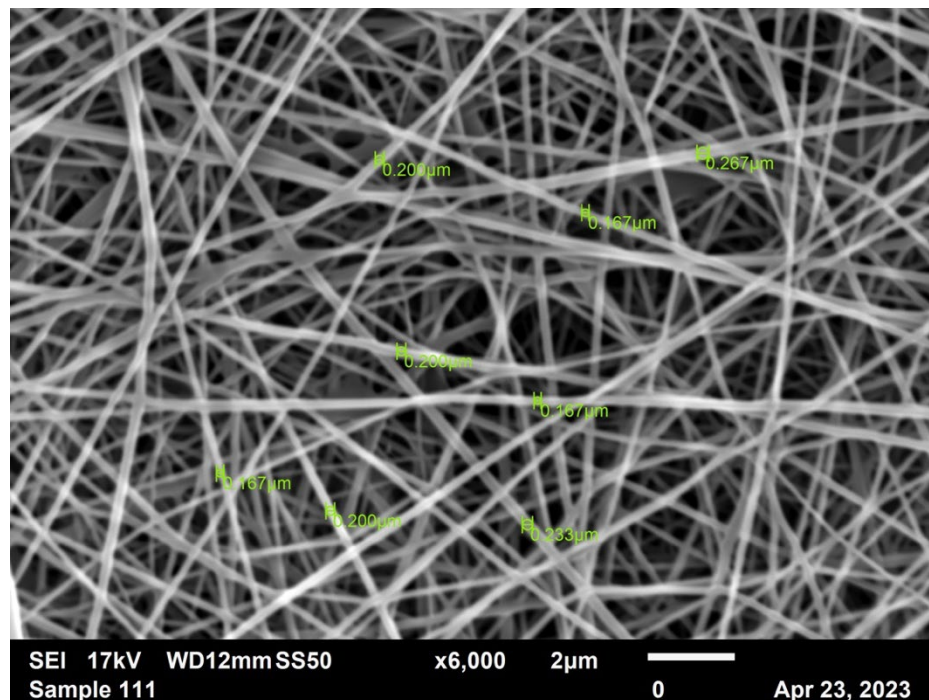


Figure 10. Nanofibers from 10 cm collection distance test with corresponding diameters.

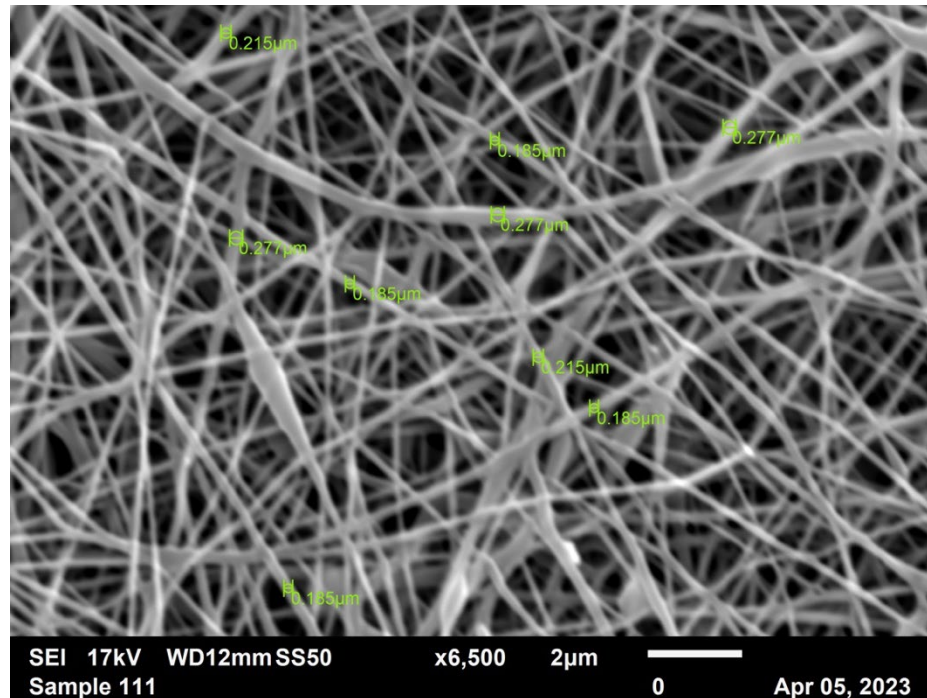


Figure 11. Nanofibers from 12 cm collection distance test with corresponding diameters.

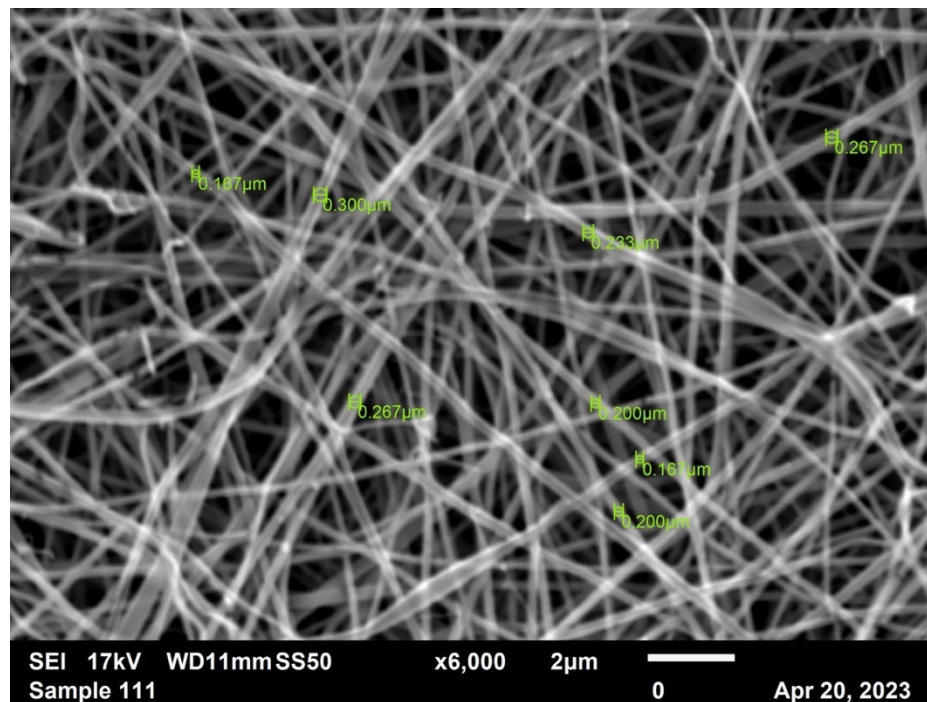


Figure 12. Nanofibers from 14 cm collection distance test with corresponding diameters.

To evaluate the nanofibers on the SEM the thickest section of each PVA mat was cut and coated with 5.5 nm of gold using a sputter coater. The mat was then placed on the SEM and four images per mat were taken for measurements, with 7-10 nanofibers per “section” or image taken at a time. Each test for each tip diameter had four images taken per sample. Table 7 shows the averages and percent differences for each corresponding collection distance.

Table 7. Evaluation of nanofibers from the Tip Diameter Variability Test

Collection Distance (cm)	Average Diameter (nm)	Percent Difference (%)
10	206.20	3.01
12	228.31	12.40
14	228.92	12.63

Evaluation

The test met the project requirement as all percent differences for each tip diameter was below 20%. The assumption made that the tip diameter would not have an impact on the diameters of the nanofibers was correct.

Conclusions

Our project, the In-House Built Electrospinning Machine, has fulfilled the promise of being able to produce nanofibers with similar dimensions to those of fibers produced in previously published experiments. Our prototype also met all our project objectives and project requirements except a schedule objective which involved having finished prototype testing by March 10, 2023. However, the team was able to get past this delay and deliver a final working prototype by April 28, 2023. A future team is expected to take on this final prototype and improve it by integrating another type of collector that can produce aligned nanofibers while maintaining the ability to interchange collector types and implementing any other useful additions or modifications.

Appendices

Appendix A – User’s Manual

Electrospinning Machine User Manual

IMPORTANT: It is necessary that the user is familiar with the capabilities and proper way of using the syringe pump, high voltage power supply, and digital multimeter before beginning the electrospinning process. Therefore, it is expected that they will read and understand the syringe pump, high voltage power supply, and digital multimeter manuals before reading this manual. An appropriate electrospinning machine setup image and voltage safety display with labels is provided below the instructions to aid the user in setting it up properly.

NOTICE: The fume hood has an air flow alarm that is activated if it is opened over a certain limit. If it is triggered, the user can mute the alarm with the button located outside the fume hood to the far-right.

1. Fill the syringe with PVA solution or any other solution (an integer value for volume is recommended) that can undergo the electrospinning process. Clean the surface and tip of the syringe with a towel so that there is no solution on it.
2. Firmly add the desired syringe tip to the syringe. (0.51, 0.61, and 0.83 mm tip diameters were tested)

3. Slowly push the syringe plunger forward until there is a small drop of solution hanging at the end of the syringe tip.
4. Wrap the entire (front and back) flat collection plate with aluminum foil. Make sure that the more reflective and shiny side of the aluminum foil is wrapped against the collection plate which leaves the less reflective side facing outward.
5. Set the wrapped collection plate on the collection plate holder with the center of the collection plate aligned with the center of the collection plate holder.
6. Switch on the fume hood light (located on left side outside of fume hood)
7. Place the syringe on the syringe pump and ensure it is firmly held by the support on the syringe pump and the pusher block is in contact with the syringe plunger. (More details on how to load the syringe properly are available in the NE-300 Just Infusion™ Syringe Pump User Manual)
8. Plug in the syringe pump power adapter and flip the switch on the back of the syringe pump down to turn the syringe pump ON.
9. Adjust the flow rate settings (1.0 mL/hr was tested for PVA solution) and check that the diameter setting is 12.45 mm (diameter setting is for HSW 5 mL syringe, more diameters available for various syringes in the NE-300 Just Infusion™ Syringe Pump User Manual)
10. Set up a timer based on flow rate and volume of solution in the syringe.
(Volume/Flow Rate = Time)
11. Turn on the multimeter (voltage safety display) to measure voltage (labeled V) and ensure that the red probe is clipped to the orange wire and the black probe is connected to the grey wire. If the multimeter does not power on, the batteries need to be replaced. (Refer to Voltage Safety Display Setup for a detailed visual)
12. The voltage reading should be zero, if it is not zero, then the high voltage power supply is most likely plugged into an outlet in which case it is recommended to unplug before proceeding. (Refer to Figure A3 for a typical voltage reading if high voltage power supply is plugged in and OFF)
13. Adjust the collection plate distance using the etching marks on the tray (10 – 15 cm were tested for PVA solution) and make sure that the collection holder is firm, and its center is aligned with the corresponding etch mark.
14. Clip the black alligator clip to the flat collection plate and ensure that the other end of the white alligator clip is connected to the black wire from the high voltage power supply.
15. The black alligator clip should be connected to the grey wire and black wire from the high voltage power supply.
16. The red alligator clip should be connected to the red wire from the high voltage power supply and the other end should be clipped to the syringe tip. (center of the syringe tip is ideal)
17. Ensure that yellow alligator clip is connected to ground and the other end is clipped to the syringe pump.
18. Refer to the Electrospinning Machine Setup to ensure all connections are correct.
19. Before connecting the high voltage power adapter to a plug-in, ensure the power supply switch is OFF and both knobs are turned fully counterclockwise.
20. Plug the high voltage power supply into an outlet.
21. Close and lower the fume hood doors.
22. Slightly open the far-left fume hood door, turn on the high voltage power supply, and adjust the voltage knob until desired voltage is displayed (30 kV was applied during testing) on the voltage safety display ($\pm 5\%$ from ideal voltage is acceptable). It is important to remember that the voltage safety display displays in units of V, but the true output voltage is actually in kV.
23. Close the left fume hood door, slightly open the middle fume hood door, and press START on the syringe pump.
24. Begin the timer that was previously set up, the electrospinning process has begun.

25. Once the timer is over, slightly open the left fume hood door and switch the high voltage power supply OFF.
26. Unplug the high voltage power supply from its outlet.
27. Open the middle fume hood door and press STOP (**same button as START**)
28. Remove the white alligator clip from the collection plate, remove the red alligator clip from the syringe tip, and take the collection plate holder out of the fume hood.
29. Carefully remove the wrapped collection plate from the collection plate holder
30. Remove the aluminum foil from the collection plate.
31. Clean any residue left from the electrospinning process using a towel.
32. Switch the syringe pump switch up so that it shuts OFF.
33. Unplug the syringe pump and turn OFF the voltage safety display (multimeter) and fume hood lights. Close and lower the fume hood if it was opened during cleaning.
34. If the user wants to characterize the nanofibers produced, seek SEM training from Dr. Steele

Electrospinning Machine Setup

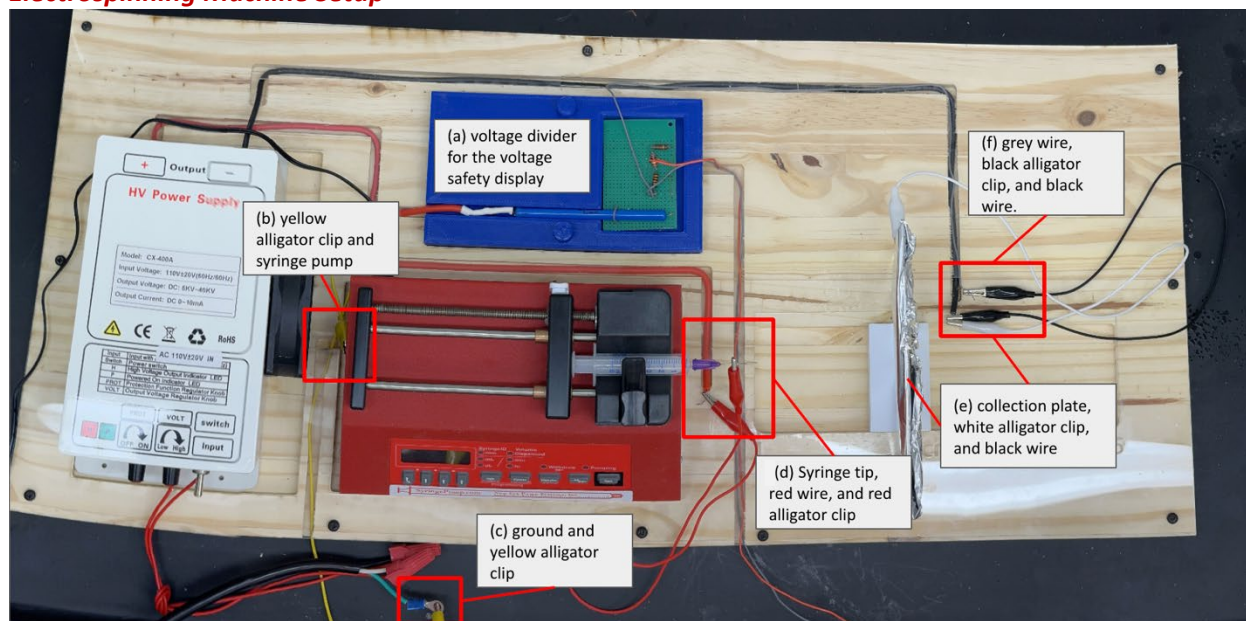


Figure A1. Electrospinning Machine Setup. (a) voltage divider for the voltage safety display. (b) yellow alligator clip and syringe pump. (c) ground and yellow alligator clip. (d) Syringe tip, red wire, and red alligator clip. (e) collection plate, white alligator clip, and black wire. (f) grey wire, black alligator clip, and black wire.

Voltage Safety Display Setup

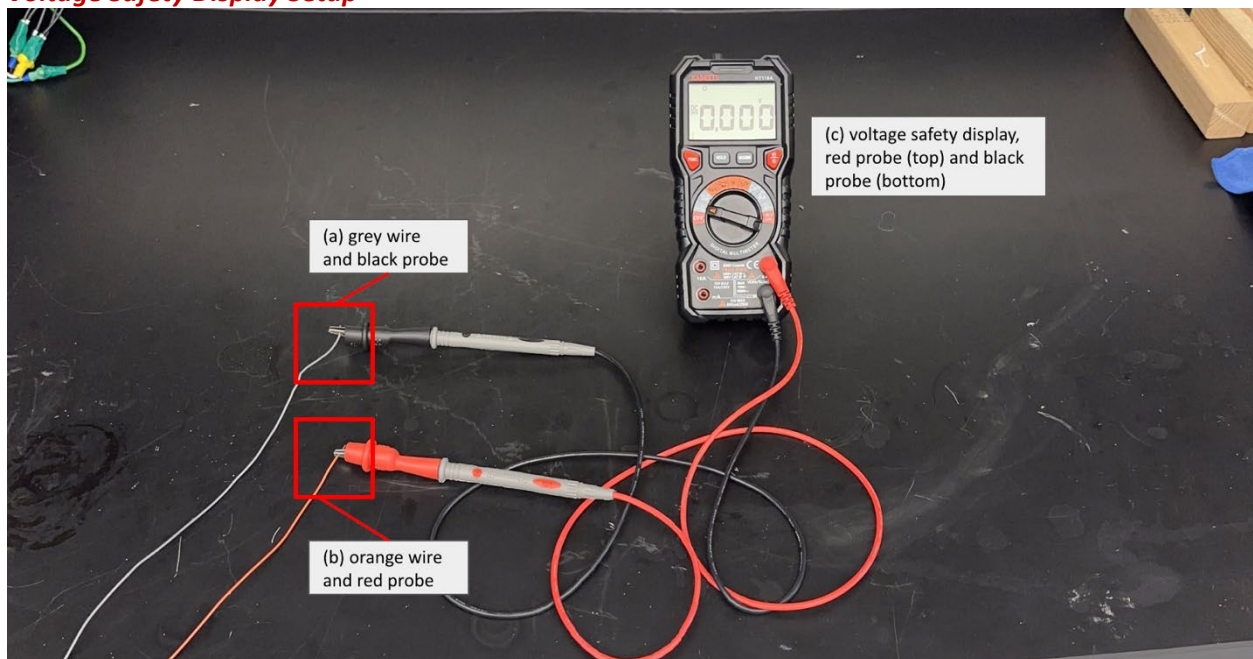


Figure A2. Voltage safety display setup. (a) grey wire and black probe. (b) orange wire and red probe. (c) voltage safety display, red probe (top) and black probe (bottom).

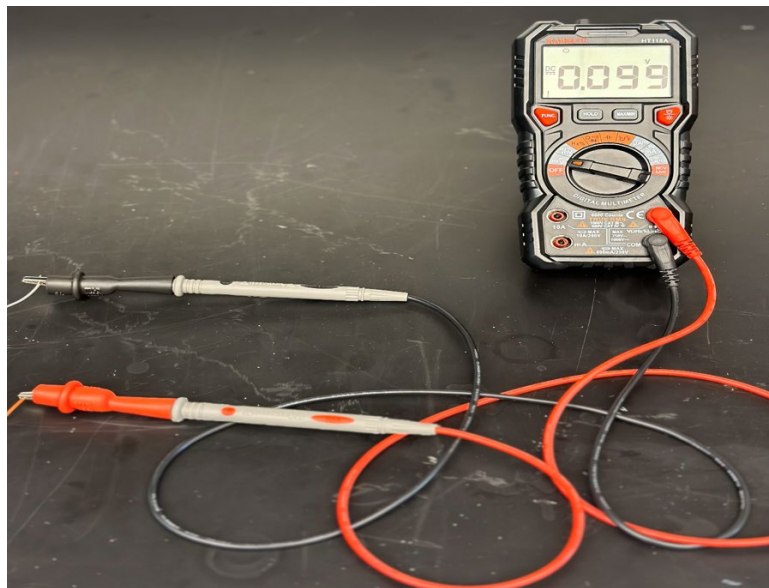


Figure A3. Typical voltage reading with HVPS plugged in and OFF (0.099 V reading is actually 0.099 kV).

Appendix B – SEM Images

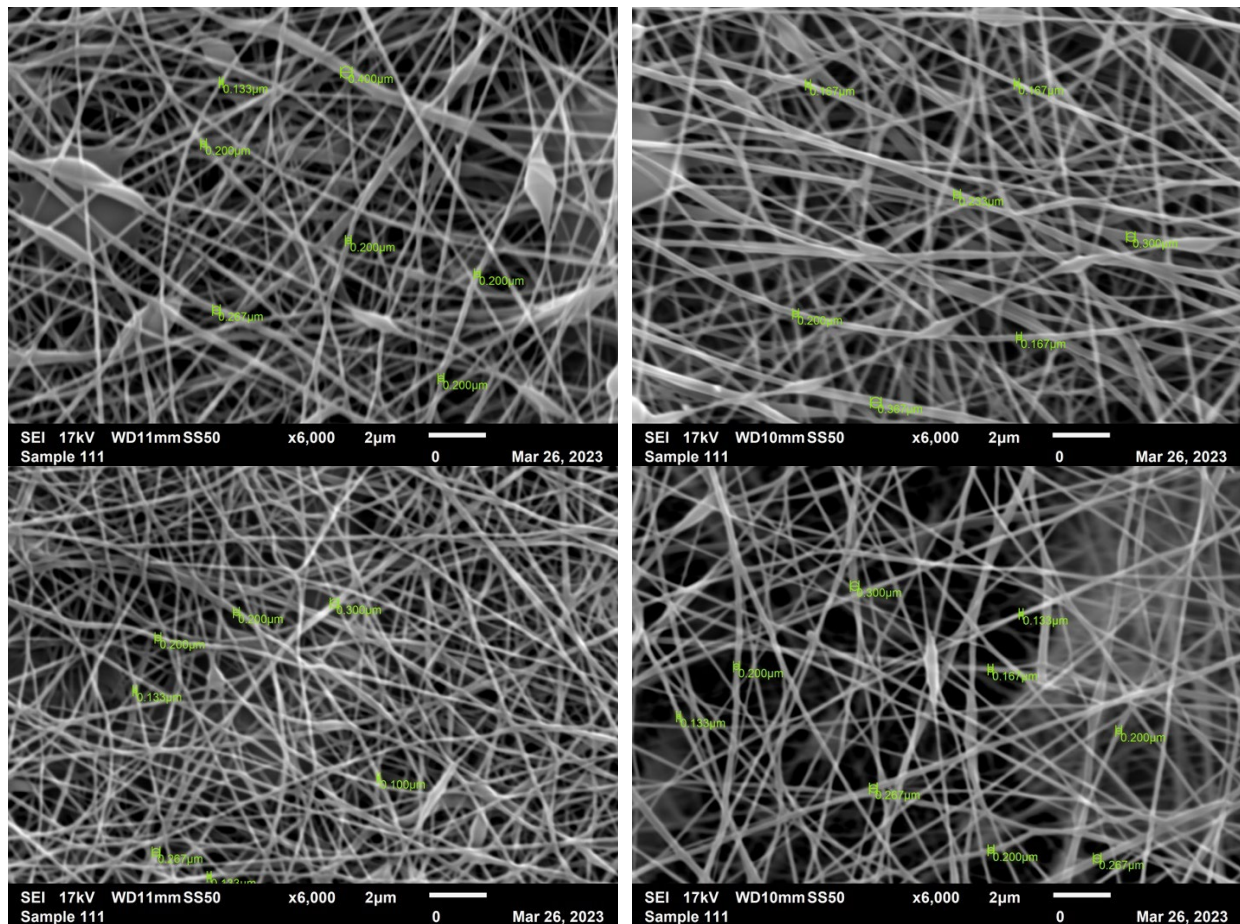


Figure B1. Proof-of-Concept #1. Conditions: 0.51 mm tip, 1.0 mL/hr, and 12 cm collection distance. Nanofiber diameters of sections 1-4.

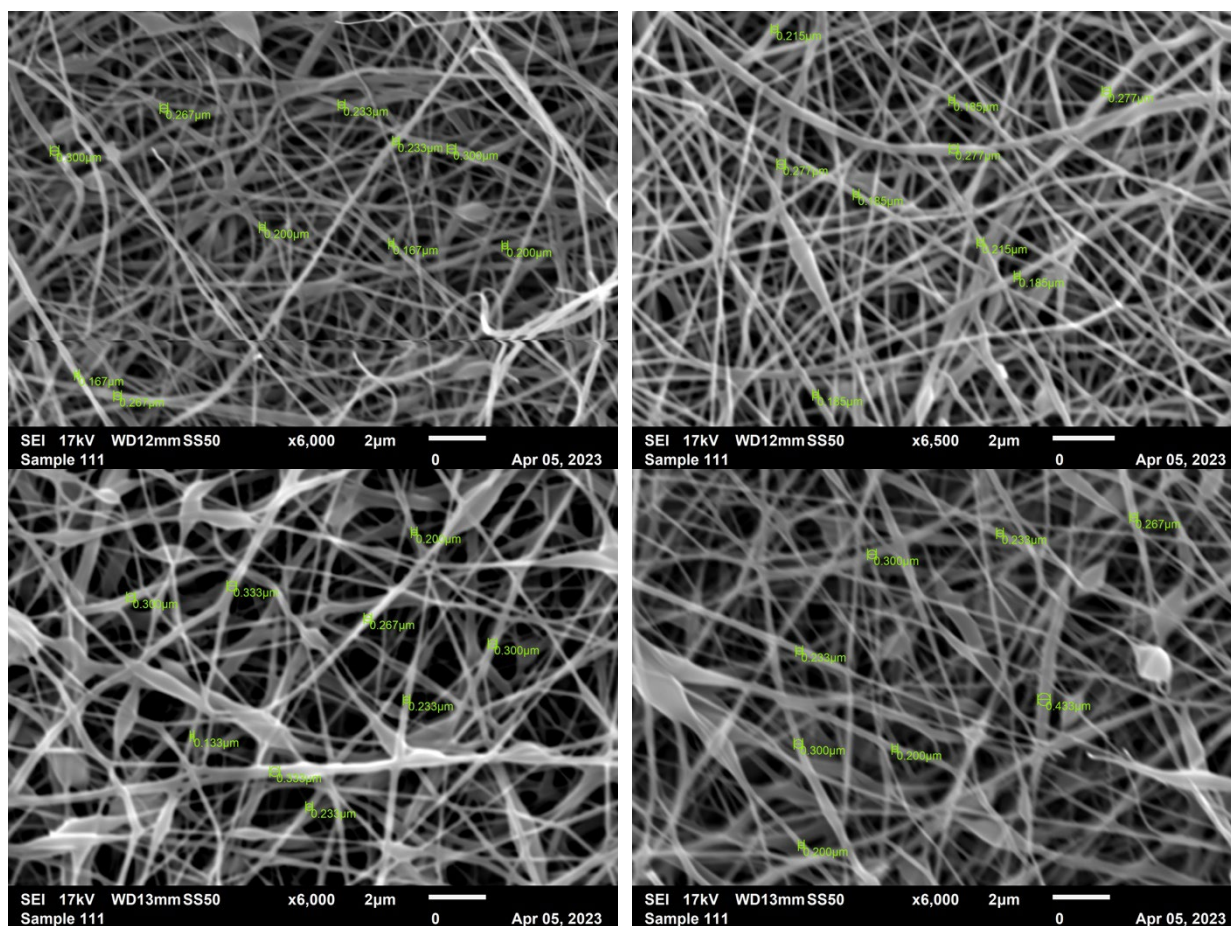


Figure B2. Proof-of-Concept #2. Conditions: 0.51 mm tip, 1.0 mL/hr, and 12 cm collection distance. Nanofiber diameters of sections 1-4.

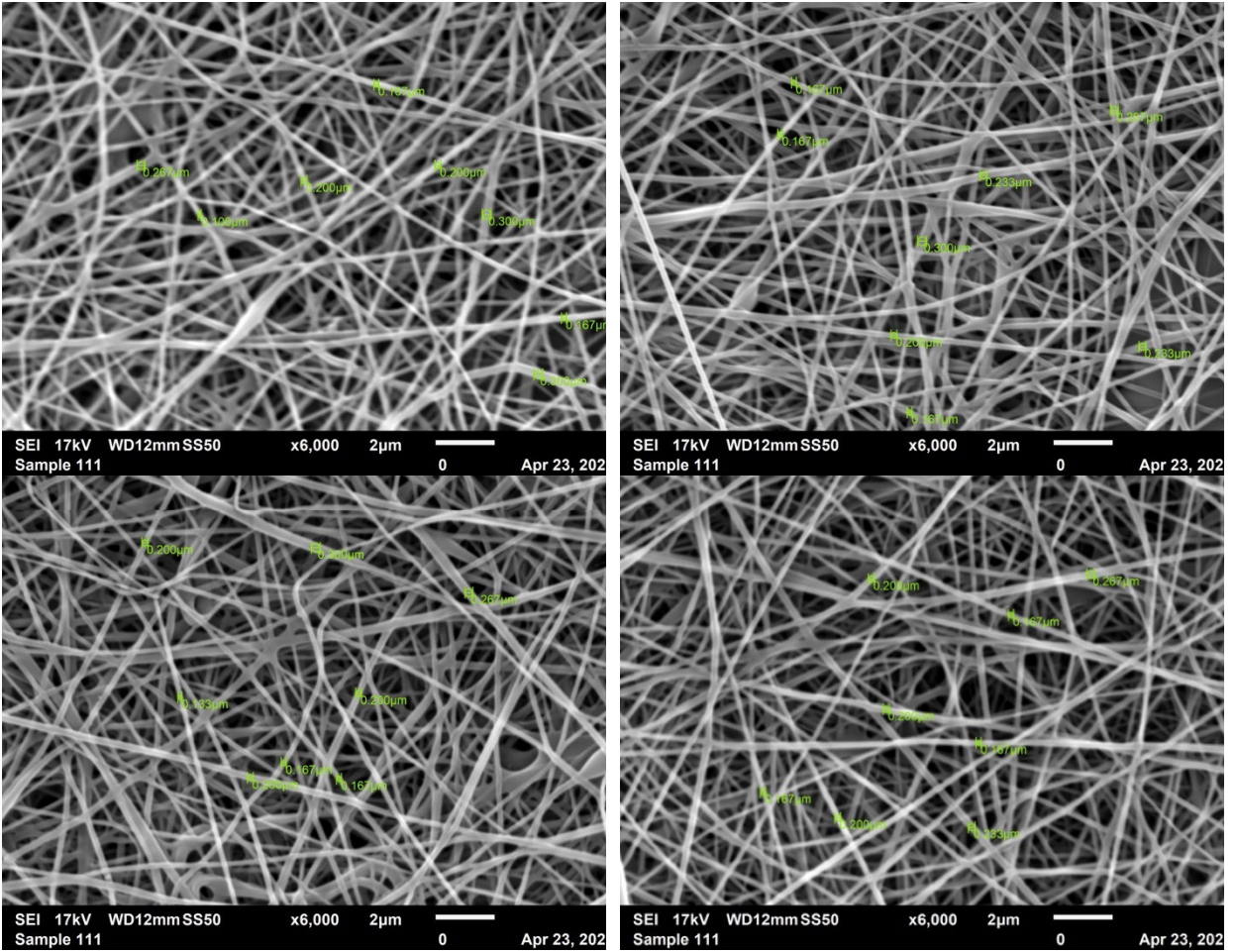


Figure B3. Collection Distance Variability. Conditions: 0.51 mm tip, 1.0 mL/hr, and 10 cm collection distance. Nanofiber diameters of sections 1-4.

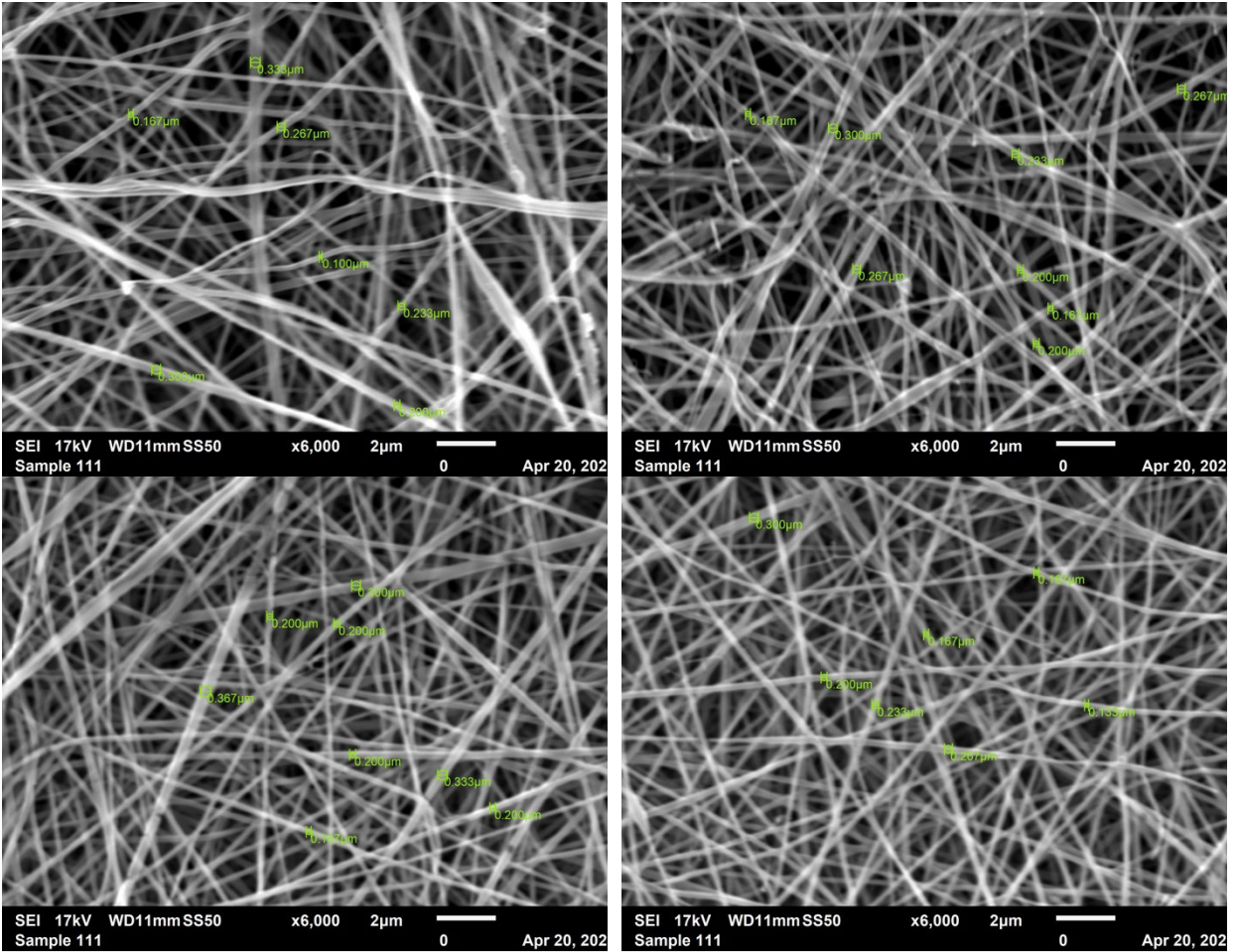


Figure B4. Collection Distance Variability. Conditions: 0.51 mm tip, 1.0 mL/hr, and 14 cm collection distance. Nanofiber diameters of sections 1-4.

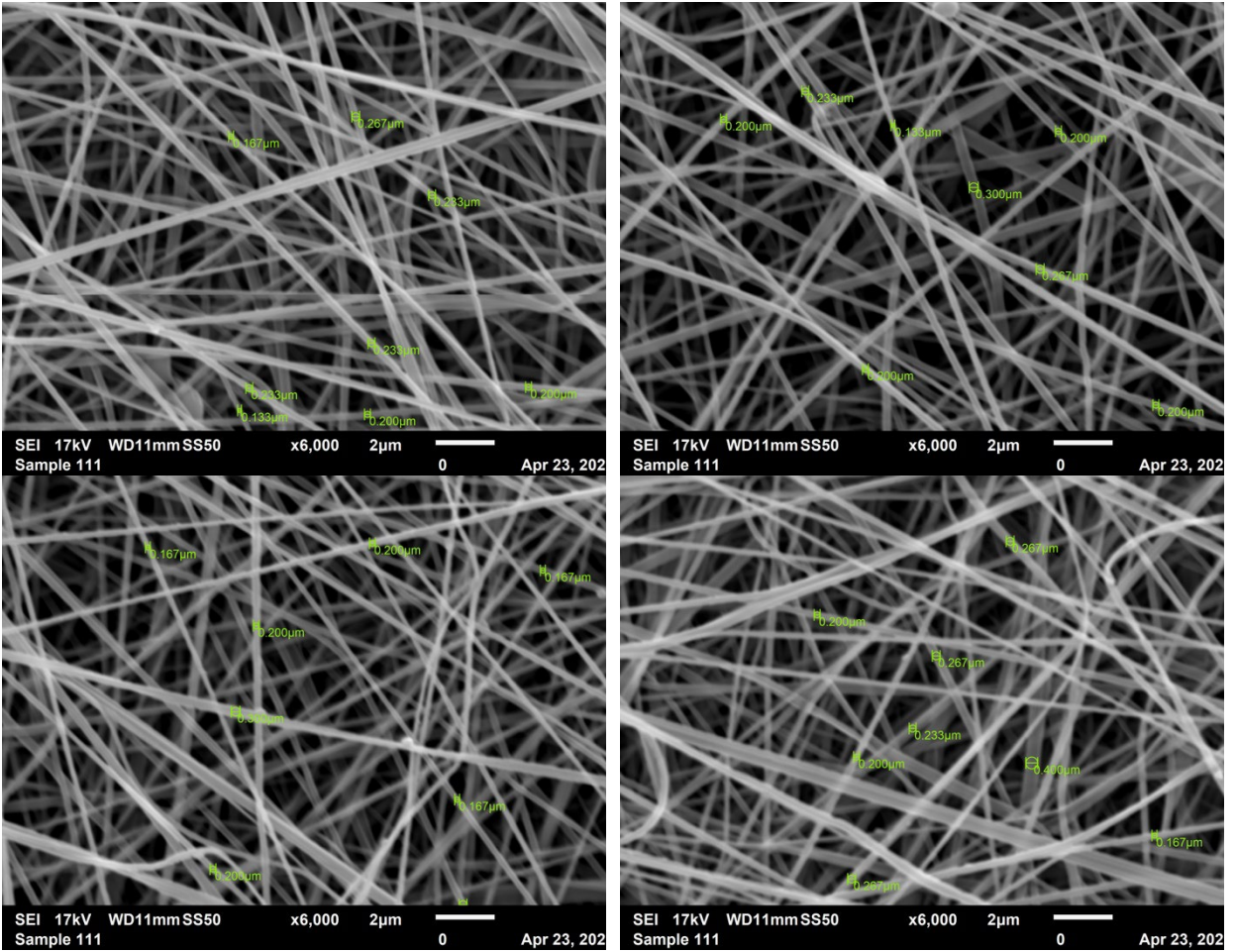
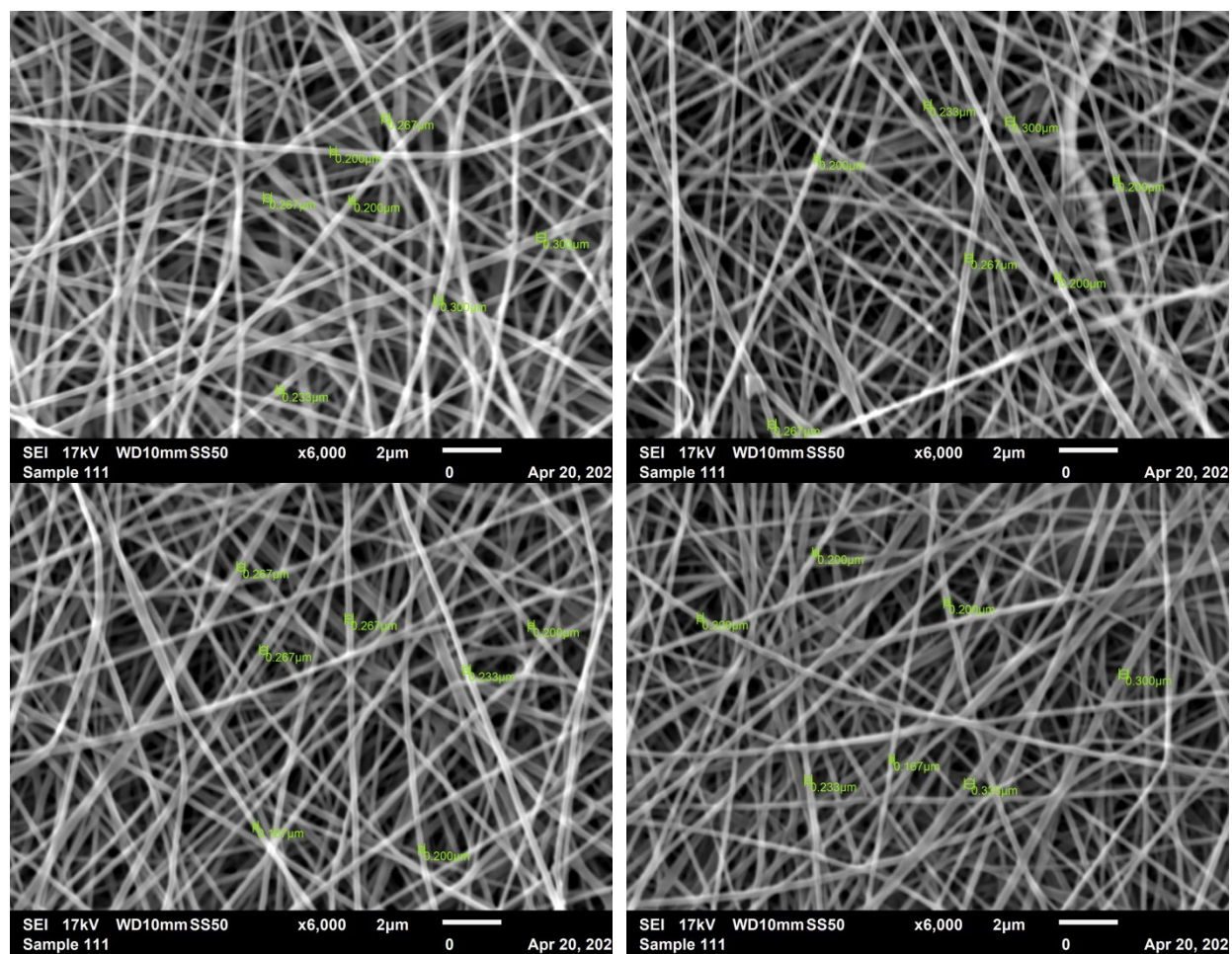


Figure B5. Tip Diameter Variability. Conditions: 0.61 mm tip, 1.0 mL/hr, and 12 cm collection distance. Nanofiber diameters of sections 1-4.



Appendix C – CAD Model of Housing Tray

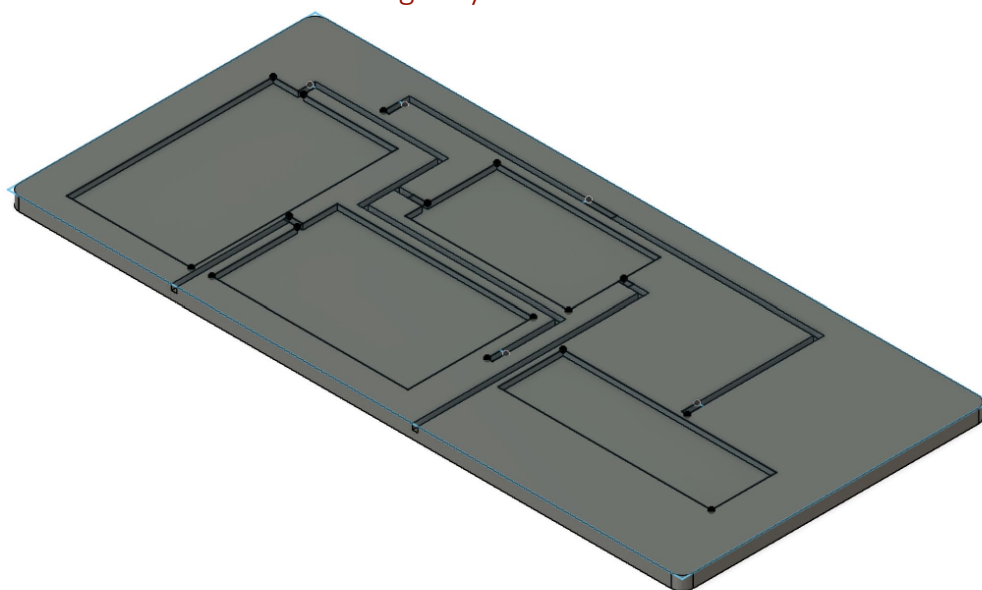


Figure C1. CAD model used to design and manufacture the housing tray.

Appendix D – Our method for creating and handling the PVA solution.

To create the PVA solution, polyvinyl alcohol of 100-200 kDa should be used to have optimal tensile strength in the final product. [3] The materials needed to prepare the PVA solution are a 100 mL beaker, conical tubes, stirrer, PVA in powder form, diH₂O water, a scientific grade microwave, a scale, and saran wrap. Below are the steps to create the PVA solution used for electrospinning.

1. Weigh the amount of PVA powder necessary for the solution (for a 100 mL solution and to create a 10% w/v solution, measure 10 mg of PVA).
2. Measure the amount of diH₂O water necessary for the solution (for a 10% w/v solution and 100 mL of solution, 100 mL of water are required).
3. Add in the measured PVA powder to the water in the beaker VERY SLOWLY and stir continuously for 5 minutes.
4. Cover beaker in saran wrap and poke holes to allow air flow. Transfer beaker to microwave and set microwave to highest setting.
5. Microwave for 30 seconds, stopping and stirring every 5 seconds.
6. DO NOT ALLOW THE SOLUTION TO BOIL, this will change the properties and concentration of the solution.
7. Allow the solution to cool in the beaker for 2 minutes, then transfer to conical tubes and allow solution to settle for at least 4 hours before using in electrospinning machine.

Appendix F – Circuit Schematics

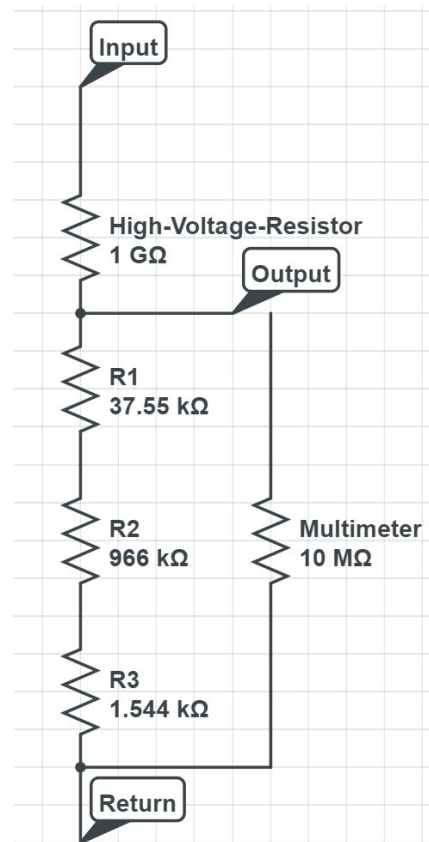


Figure F1. The circuit schematic of the voltage divider using the measured resistor values.

Appendix G – Bill of Materials

Table G1. Bill of materials

Qty	Manufacturer	Source	Part Number	Part Description	Cost per Unit (\$)
1	CXDZ	AliExpress	CX-400A	High voltage power supply, 5 - 40 kV output	81.50
1	New Era Instruments	Scientific Instruments	E46/NE300US	Just Infusion one channel syringe pump	295.00
1	Uxcell	Amazon	B01IX44S0W	Electric copper core flexible silicone wire, 22 AWG, 40kV	16.49
1	Ohmite	Digi-Key	MOX-4N-131007 JE ND	1 GOhms $\pm 5\%$ resistor, axial high voltage	26.45
1	Swaytail	Amazon	B096S2JXYW	Alligator clips, 10pcs	4.99
1	Metcal	Techni-Tool	920100-TE	Stainless steel precision needles, 1" tip, 20 gauge, 50 pack	28.80
1	Metcal	Techni-Tool	921100-TE	Stainless steel precision needles, 1" tip, 21 gauge, 50 pack	28.80
1	Metcal	Amazon	918100-TE	Stainless steel precision needles, 1" tip, 18 gauge, 50 pack	9.73
1	Insignia	Best Buy	5986103	Surge protector, 8 outlets, 600J	24.99
1	Insignia	Best Buy	6465025	High-output universal AC adapter, 3A	24.99
1	R-Tech	Amazon	B00FEOB4EI	Power adapters, 12V, 1A, 5 pack	17.99
1	KAIWEETS	Amazon	B07SHLS639	Digital multimeter TRMS 6k counts voltmeter	27.59
1	HANDSKIT	Amazon	B074L1NXRX	Electrical multimeter test leads set with alligator clips, 1kV, 10A	16.99
1	-	Home Depot	812567011753	Laminated Spruce Panel	25.87
1	-	Home Depot	185435000884	Laminated Pine	21.98
2	OPTIX	Home Depot	769125010614	Clear Acrylic Sheet	38.38

Appendix H – Codes & Standards

NFPA 70 E: Electrical Safety in the Workplace 2013 Edition. In *NFPA National Fire Codes*. [Online]. Available: <http://codesonline.nfpa.org>

United States Department of Labor. *Law and Regulations /Occupational Safety and Health Administration*. [Online]. Available: <https://www.osha.gov/laws-regs>.

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[1] M. K. Leach, Z.-Q. Feng, S. J. Tuck, and J. M. Corey, "Electrospinning Fundamentals: Optimizing Solution and apparatus parameters," *Journal of visualized experiments : JoVE*, 21-Jan-2011. [Online]. Available: <https://www.ncbi.nlm.nih.gov/pmc/articles/PMC3182658/>. [Accessed: 02-Oct-2022].

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[3] N. H. Akhmal Ngadiman, M.Y. Noordin, A. Idris, A. S. Abdul Shakir, D. Kurniawan, "Influence of polyvinyl alcohol molecular weight on the electrospun nanofiber mechanical properties," *2nd International Materials, Industrial, and Manufacturing Engineering Conference*, 4-Feb-2015. [Online]. [Accessed: 13-Nov-2022].