Development of precision pump and high voltage DC-regulator for electrospinning apparatus: experimental test with preparation of PVA microfiber

F Faizal\textsuperscript{1,2}*, A M Al-Fikri\textsuperscript{1}, A Abdurrochman\textsuperscript{1}, I M Joni\textsuperscript{1,2} and C Panatarani\textsuperscript{1,2}

\textsuperscript{1}Department of Physics Faculty of Mathematics and Natural Sciences, Universitas Padjadjaran, Jl. Bandung-Sumedang km. 21, Sumedang Regency, West Java, Indonesia 40363

\textsuperscript{2}Nanotechnology and Graphene Research Center (PRINT-G), Universitas Padjadjaran, Jl. Bandung-Sumedang km. 21, Sumedang Regency, West Java, Indonesia 40363

*ferri.faizal@unpad.ac.id

Abstract. A digital "micro-controller-based" electrospinning apparatus was developed. The system mainly used a precision pump equipped with 20 kV and a high voltage DC regulator utilizing a used flyback transformer. Electronic circuit and power blocks evaluation has shown the linear characteristic. The setting parameters such as flow rate and total volume were calibrated by using a measuring-cylinder and a digital stopwatch. The result showed a linear trend, verified by the correlation coefficient between given and measured flow rate at $R \approx 1$. The precision pump can operate at 0.5 ml/hour (8.3 micro-liters per minute) using the 12 ml standard syringe. An 0.5 mm inner diameter needle was used in the integrated experimental and performance test. The testing material was the polyvinyl alcohol (PVA) solution at ambient condition (room temperature and standard pressure). At fix 12 cm distance between electrodes, one hour experiment, and flow rate variation. Some experimental tests resulted in membranes contained 36-61 microns diameter of stacked PVA fiber. Finally, the microfiber diameter distribution was analysed using image processing software. The result showed the dependencies of the size distribution to the flow-rate and applied voltage.

1. Introduction

Electrospinning is a method for preparing micro to nanoscale fiber. It was one of the most popular techniques on fiber preparation due to the simple, effective, and has a potential extension to produce on a large scale via multiple jet spray [1]. The material prepared by the electrospinning method was ranged from polymer, composites [2, 3], immobilized enzyme [4], to various ceramic metal oxide [5, 6]. The fiber made by electrospinning was known for having the complex dependencies on the different experimental parameters. A previous study [7] classified the parameters into three distinctive classes: environmental parameter, material parameter, and the electrospinning parameter. Environmental parameters are humidity and temperature; these parameters also valid for the closed environmental system (e.g. chamber). Material parameters include precursor concentration, electrical conductivity, viscosity, solvent volatility, molecular weight, and molecular structure. The experimental (electrospinning) parameters are spray distance, applied high voltage, the flow rate of the precision pump, and vessel or needle diameter.

In this study, the experimental parameter of the electrospinning process will be revisited through
the step by step design, development and testing of a precision pump and high voltage power supply (HVDC) as the major important device to control the experimental parameter. The analysis was focused on the most affecting parameters, which affect fiber diameter distribution. The parameters are applied high voltage, and the flow rate of the liquid precursor. Previous reports presented the scaling law of fiber diameter as the function of the mass spinning throughput and electrical current [8,9]. In another experimental study [10], the experiment resulted in micron order fiber diameter (using PAN fiber) by applying 10-20 µl/min flow rate at 10 cm distance. Another study reported that controlling the constant current may lead to very precise size distribution and morphology [9]. However, measuring or detecting nano- pico ampere order of electrical current with high precision is difficult. So, stable voltage control was chosen in this study due to the simplicity.

Two primary devices typically used in the electrospinning apparatus was designed and implemented: a precision pump (in the form of a syringe-type pump) equipped by an open-loop flow rate controller and the HVDC power supply developed from a used flyback transformer. Stable performance of flow rate (mass spinning throughput) and high voltage output will be tested via experiment on the preparation of polyvinyl-alcohol (PVA) microfiber.

2. Experiment

2.1 Precision pump design and calibration

The typical schematic setup of the electrospinning apparatus was depicted in figure 1. Another experimental type might be performed in a vertical formation, such as reported somewhere [11]. As mentioned earlier, there are two developed components involved: a precision pump and a high voltage power supply (HVDC). A simple syringe-type pump for controlling the microliter scale flow rate was designed by utilizing thread to convert the rotational motion of motor stepper into translation shifting and pushed the syringe at the specific rate. One of the critical considerations in designing the flow controller is smooth translational motion.

![Figure 1. Horizontal scheme of electrospinning experiment.](image)

The flow rate controller was consists of a pusher-thread motor stepper (mechanical section) and an open-loop controlling circuit (electronic part). At the open-loop controlling circuit, the setpoints (flow rate or volume) were assigned by the user from the keypad. The setpoint data then passed to the microcontroller (ATmega328p [12]) and based on those assigned values (either volume or flow rate), the rotation, and translation step were decided. The selected flow rate or volume data are displayed in the LCD. A complete circuit board of the controlling system with the detailed port is presented in figure 2. The translational rate was calculated in the microcontroller via the formula
\[ R_s = \frac{P_{\text{thread}}}{N_s} \]  

where \( R_s \) is the rate of translation per step, \( P_{\text{thread}} \) is the thread pitch and \( N_s \) is number of step at full rotation. In the mechanical pump section, conversion from rotational to translation motion utilizes the thread with 2 mm pitch and 2 mm lead. The thread has driven by motor stepper (NEMA 17HS4401 Hybrid) equipped by motor driver (DRV8825) with 1.8° resolution (the complete board was displayed in figure 3). This mean for one full thread rotation (360°) there are 200 steps and the calculation using equation 1 resulting 0.01 mm/step translation rate. The motor stepper controlling board and stepper driver was presented in figure 2 and figure 3.

**Figure 2.** Digital circuit board for controlling the motor stepper, applied for precision pump.

A series of experimental test was carried out for calibrating the input values (flow rate or volume) to fit with the desired output values. A 12 ml syringe and 0.5 inner diameter needle were placed in the precision pump. The measuring cylinder was used to measure the volume of outflow liquid (water and PVA 10 %). The flow rate under the calibration process was varied from 0.5 to 5 ml/hour until the outflow volume reaches 1 ml. The outflow was measured with 0.1 ml resolution, and the time was measured with a stopwatch with 0.005 s resolution. Then, the volume calibration was held to measure the linearity of the volume output versus the volume input. The flow rate was then kept at 5 ml/hour with pumping time at 12 minutes.

**Figure 3.** Motor stepper driving circuit (rotation driver).
2.2 HVDC power supply design and test

In order to apply electrical forces on the outflow liquid precursor, a high voltage power supply (range 10-20 kV) was designed and implemented. The positive electrode of the power supply was applied on the metal vessel (spinneret/needle) to modify the spherical outflow menisci to the shape of the cone (Taylor cone). A used flyback transformer was utilized in the generator circuit. The schematic circuit of the flyback transformer is presented in figure 4.

From the figure 4, the circuit is obviously described a simple push-pull oscillator. Input DC voltage is applied to the transistor and feedback simultaneously. In practice, the transistor Q1 and Q2 will not turn on at an exact similar time due to their slightly different characteristic. At this stage, there always one transistor at the off state, and the other state is on. For an example, if Q1 is on, the current would flow in the coil feed to Q1 basis, then the current will pass through the primary coil connected to Q1 and Q2 is off. Magnetic flux in the transformer core would increase versus time. At a certain time, it would be saturated, and flux magnetic reached the maximum. At this stage, all voltage applied to the coil would drop and reversed the polarity (Q2 conducted and Q1 cut off). This oscillation voltage will be continuously directed to the primary coil resulting in high voltage at the secondary coil. The output voltage then applied to the rectifier to obtain DC voltage.

![Figure 4. High voltage generator circuit utilizing oscillator and flyback transformer.](image1)

![Figure 5. Digital circuit board to control the input voltage and display the output voltage applied to the high voltage generator.](image2)

The experimental testing of the HVDC power supply was carried out to analyze the linear relation of output voltage versus input voltage. During calibrating the voltage display and measurement range, a high voltage probe was used as the reference. The probe has DC voltage range at 0 – 30 kV with 1 kV resolution.

The HVDC power supply circuit are presented in figure 5. The circuit takes 0-12 Volt DC input mapped into 0-20 kV high voltage while the output displayed was calculated from the input voltage. Some series of data calibration was taken to determine the linearity of the input vs output voltage. The resulting equation was used to display high voltages values in four series of seven segments.

2.3 Integrated electrospinning system and test performance

The developed precision pump and HVDC power supply were integrated into an electrospinning system as schemed in figure 1. The setup used in performance tests by the preparation of polyvinyl alcohol (PVA) microfiber. The system was set horizontally with the fixed distance between needle and collector at 12 cm. A needle was used as the vessel/spinneret with an inner diameter of 0.5 mm. The parameters were flow rate at 0.5 ml/hour and applied voltage (12 kV and 18 kV). Afterward, the flow
rate was increased to 1 ml/hour at 12 and 15 kV. The experiment was conducted in ambient pressure and temperature until the injected volume reaches 1 ml.

Applied high voltage causes a strong electrostatic field and induce the charge in the surface of the liquid phase precursor. The increasing number of charge at the same signs causes the repulsion and shear stress on the surface of the fluid. These repulsive forces have opposite directions with the surface tension create the change in spherical menisci into a conical shape (Taylor cone). At the critical voltage \( V_c \), the forces balance was disturbed and caused the charged jet to appear from the tips of the conical shape. The expression of the critical voltage is written as follow [11]:

\[
V_c^2 = 4C \frac{l^2}{r^2} \left( \ln \frac{2l}{r} + 1.5 \right) (1.3\pi r) \tag{2}
\]

Equation 2 shows the relation between the critical voltage \( V_c \) and the tip to collector distance \( L \), the length of fluid column \( l \), the inner radius of the vessel/needle (spinneret) \( r \) and surface tension \( \gamma \) and proportionality constant \( C \) which was determined empirically \( (C = 0.09) \) [11]. The equation can give a clear relation while balancing the distance and voltage to obtain a stable cone-jet.

The resulted fiber then analyzed using an optical microscope, and the fiber photograph was analyzed using an image processing software (ImageJ), to obtain fiber diameter distribution. From the distribution, the effect of the experimental parameter to the size distribution will be discussed.

3. Result and Discussion

3.1 Calibration of precision pump

As explained in the experimental section, the precision pump was calibrated using a measuring cylinder for volume measurement and supported by a stopwatch for measuring flow rate. The evaluation detail and test for the precision pump component are not shown here, but the adjustment to open-loop control parameters was made to fit the calibration process. The calibration test collected the data of output volume (from measuring cylinder) vs. input data volume (from the pump display). The plot of this data was presented in figure 6.

Every data point in the plot in figure 6 was an average of five times repeated measurement. The standard deviation (error bar) cannot be seen on the graph due to their small values (negligible). The data has shown a linear fit with correlation coefficient \( R \approx 1 \), line gradient equal to 1, and zero offsets (from the fitting calculation).

![Figure 6. Volume calibration data of the developed precision pump.](image)

![Figure 7. Flow rate calibration data of the developed precision pump.](image)

The data in figure 7 shown the plot between output flow rate data (from measuring cylinder and stopwatch) versus input flow rate data (as displayed from pump display). Since the timing of the
output was measured by stopwatch with 1 ms resolution) and the input timing was measured by the internal clock of micro-controller (higher resolution), there may be the propagation of indirect measurement error of the output data. As shown in the data, the error bar is still negligible, but the gradient was not equal to 1 anymore, and it was clear that the offset was also not zero (figure 7). From the graph, the full range of the flow rate setting for this precision pump was 0.5 - 5 ml/hours. The liquids used as the test sample in this experiment are water (black dot) and 10% wt PVA solution (red dot). The linear properties are observed in both graphs in the graph, the full range of the flow rate setting for this precision pump was 0.5

Figure 8. High voltage DC output versus low voltage input and the conversion formula obtained from the developed HVDC power supply.

Figure 9. Fiber diameter distribution and morphological structure (inset) of PVA 10 wt% with:(a) flow rate = 0.5 ml/hour with V = 12 kV, (b) flow rate = 0.5 ml/hour with V=18 kV, (c) flow rate = 1 ml/hour with V = 12 kV and (d) flow rate = 0.5 ml/hour with V=18.
3.2 Calibration of HVDC power supply

The evaluation was made to analyze the relation of input data (low voltage) with the output data (high voltage). The plot of the data was presented in figure 8. It is shown that the proportional pattern with some non-linearity observed in several regions. The resulted HVDC power supply could be operated in the 2-20 kV range.

The linear regression calculation resulted \( V_o = 1.57V_i - 0.00072 \) with \( R^2 = 0.97 \). The largest value of error was observed at the 3.5 V input voltage, which shows the extreme point in the curve. This might be caused by the spike during the voltage increment; this result also indicates that at this extreme point, the flyback transformer was unstable. The stable region was reached in the region with more than 4V input. The linear equation obtained from the regression calculation was made into the calibration formula and programmed into the microcontroller to be displayed in the built-in screen.

3.3 Performance test with PVA microfiber preparation

As mentioned in the experimental section, the PVA fiber was divided into several variations of treatment. In this study, the treatments are flow rate and voltage, while the distance and vessel size was fixed. The observation was started at the condition of the output precursor reach the cone-jet state. To obtain the jet, the tuning of flow rate and voltage was conducted simultaneously.

From the observation at 0.5 ml/hour (8.3 microliters per minute), the stable cone jet was started to appear at 12 kV. The fiber was formed at the end of the tips. Below this voltage, the large droplet was present due to insufficient electrostatic forces. The stable cone was observed at the flow rate of 1 ml/hour. The experiment was conducted for several different values of parameters such as flow rate: 1 ml/hour with 12 kV and 15 kV, and flow rate at 0.5 ml/hour with 12 kV and 18 kV.

The result sample was characterized using a microscope and the diameter of the fiber then measured by an image processing software. The data were grouped into the histogram (size distribution). The diameter distribution was shown in figure 9. It was obtained from the photomicrograph on each inset. Figure 9(a) shows the fiber size distribution for the electrospinning with a 0.5 ml/hour flow rate at 12 kV. The average values from this distribution were 41.48 ± 8.99 \( \mu \)m. The constant 0.5 ml/hour flow rate was fixed, and the voltage was increased to 15 kV. Unfortunately, the result of this condition was failed to capture, and the causes of this problem still unknown. Hence, the voltage was skipped to 18 kV, to know the tendency of the increased voltage to the size distribution. The 0.2 ml/hour resulted the fiber size distribution presented in figure 9(b). The average value was 36.90 ± 7.00 \( \mu \)m, and this size was significantly decreased compared to the lower voltage result (12 kV).

If the flow rate was increased to 1 ml/hours, then as it has been suspected, the average size of the fiber would change significantly in diameter. This phenomenon was presented in figure 9(c) where the sample was applied with 12 kV, and the result shows 48.07 ± 12.93 \( \mu \)m average fiber diameter. Here, the widening of the distribution was indicated by the increasing value of the standard deviation. As the voltage increase to 15 kV with the fixed flow rate (1 ml/hour), the average value of the fiber diameter was decreased to 44.97 ± 7.12 (figure 9(d)).

Overall, the obtained histogram was fitted by the gaussian curve and summarized in figure 9(e). From the graph, it was observed that the fiber diameter has a tendency to decrease at the increasing voltage and increase at the increasing flow rate. This result was typically observed in the electrospinning experiment. It also can be explained qualitatively from the force balance of fluid and electrostatic point of view [11].

The fiber diameter resulted in these experiments were still in the micron order. In order to decrease the diameter into the smaller size distribution, the size of the vessel should be reduced, and the evaporation rate in the experiment should be controlled. Another approach is to use the polymer (PVA) as the template for the main material to be formed as fiber. Quantitative adjustment, such as material ratio, can be used to control the morphology and size distribution of the prepared fiber [13].
4. Conclusion
Based on the result, the electrospinning system was developed. The system consists of the precision syringe pump, and the high voltage power supply. The system was designed and implemented with full functionality to perform electrospinning preparation of the microfiber. The precision pump can be operated in a 0.5-5 ml/hour range with 0.5 ml/hour resolution using an open-loop controller. The control method implemented in the controlling circuit and mechanical section, while the HVDC developed in this study works at 2-20 kV range with 1 kV resolution driven by an open-loop analog circuit.

The experimental test at several sampling conditions demonstrated the ability of the system to control the fiber size distribution of PVA via flow rate and applied voltage. Some improvements in the experiment are still needed to obtain fine fiber diameters, such as decreasing the vessel diameter or increasing distance with a controlled evaporation rate. The future application may extend to achieve not only polymer microfiber but also another material such as metal oxide, and carbon fiber by using the polymer as the template.

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