Effects of Electrospinning Parameters on Carrageenan-Based Nanofibers Fabrication

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***Abstract*:** Polymer nanofibers are known to exhibits excellent properties that made it very useful in producing and development of tissue engineering, filtration, biochemical engineering and many more. Nowadays, electrospinning is the most commonly and preferably used to manufacture these polymer nanofibers because the nanofibers produced using this technique ably to produce a high surface area to volume ratio. Carrageenan-based nanofibers were prepared by electrospinning of polyvinyl alcohol (PVA)/carrageenan (CAR) solution. The solution is made of 1 w/v% of CAR solution and 13 w/v% fully hydrolyzed PVA solution. The solutions were mixed with a ratio of 70:30, creating the polymer solution, FH PVA/CAR solution. The morphology and diameters of the nanofibers were observed by using a scanning electron microscope (SEM), the contact angles on the nanomembrane were measured by static micro-drop observation, and the effects of electrospinning parameters including voltage, distance between tip of needle and collector, as well as the flow rate of the solution were observed. The study was carried in a series of experiments involving different voltages, flow rate and tip to collector distance. The different voltages include 9kV, 12kV, 15kV and 18kV where the average diameter is observed to be increasing from 160.94 nm, 137.95 nm, 128.18 nm, to 119.69 nm, respectively. This shows that by increasing applied voltage, the fiber diameter decreased. it is also observed that the mean fiber diameter is 129.52 nm, 133.85 nm, 138.73 nm, and 140.49 nm, accordance with the flow rates 0.2 ml/h, 0.3 ml/h, 0.4 ml/h, and 0.5 ml/h. this implies that increment of flow rate causes the increment of fiber diameter. On the other hand, the effect of increasing tip-collector distance from 12cm, 15cm, 18cm, to 21cm resulted in decreasing of fiber diameter that is 132.94 nm, 131.79 nm, 124.38 nm, and 114.72 nm, respectively. Contact angle of the electrospun nanofibers decreases over time to below $9^{0°}$, showing that the nanofibers is hydrophilic. This study is helpful to optimize the electrospinning process parameters for the preparation of PVA/CAR nanofibers whether according to desired mean diameter.

***Keywords*:** Carrageenan, Electrospinning, Parameter Effects, Polyvinyl Alcohol.

I. INTRODUCTION

Carrageenan (CAR) is a species of seaweed that is belonged to the marine red algae family. It is considered to be the most appealing out of the other sources of the polysaccharides, with different type of structures and functional properties, CAR is known to own gel-forming ability, biocompatibility, abundance and renewability [1]. Carrageenan can be categorized in to six different basic forms which are Kappa (κ)-, Lota (ι)-, Lambda (λ)-, Mu (μ)-, Nu (ν)-, and Theta (θ)-carrageenan [2]. They are classified accordingly to the degree of substitution that occurs on their free hydroxyl groups [3]. (κ)-, (ι)-, and (λ)-carrageenan are known to be the most important forms out of the basic forms of carrageenan commercially due to their viscoelastic and gelling properties [3]. (κ)-carrageenan has only one negative charge per disaccharide with a tendency of forming a strong rigid gel [4]. The carrageenan that will be used in this study is (κ)-carrageenan. Carrageenan is commonly used as thickening, emulsifying and also its stabilizing agent usually used in the food and pharmaceutical administration as well as industrial applications [5]. CAR is also used in tableting excipients due to its great compatibility, high robustness and persistent viscoelasticity of the tablet during compression [6]It is also known that carrageenan have the potential of having the properties of anticoagulant, anticancer and modification on the immune system which may be very important in the pharmaceutical industry [4].

Polyvinyl Alcohol (PVA) is known to be a water-soluble, hydrophilic semi-crystalline, and non-toxic synthetic polymer, widely used in the production of films and fibers as well as the applications in filtration and biochemical engineering [7]. This is due to its amazing chemical stability, mechanical and physical properties [8]. PVA is one of the most highly biocompatible and non-toxic polymers making it a widely used polymer in film forming, electrospinning antimicrobial nanofibrous mat to be used in different applications, such as medical, cosmetic, food, pharmaceutical, and packaging industries [9]–[17]. PVA can be categorized into two types: fully hydrolyzed PVA and partially hydrolyzed PVA. In this experiment, fully hydrolyzed PVA will be used and blended with selected CAR in order to form the desired electrospun nanofibers.

The electrospinning process is a process that is electrostatic driven that is based on high electric field in order to produce any polymer-based nanofibers or nanostructures [18]. Electrospinning offers a distinct advantage of producing nanofibers in the micro- to nanometer range of fibers with high surface area to volume ratio compared to conventional fibers [19] made by melt spinning [20], wet spinning [21], and extrusion molding [22]. Any type of nanofibers or nanostructures can be obtained by using or mixing different types of polymer or bio-polymer [23]. In order to obtain different structures of nanofibers, the properties of the polymer solution or the amount of electric field can be optimize according to desired nanostructures. There are several other parameters that can be conducted during electrospinning process to serve such desire including the distance between the needle and the collector, flow rate, and concentration of the polymer solution. This study will help to outline the effects of the electrospinning parameters to the fabrication of the nanofibers.

II. METHODOLOGY

A. Materials

The (κ)-CAR was used as the natural polymer source while the fully hydrolyzed PVA were used as the synthetic polymer source. The (κ)-CAR used was a semi refined carrageenan purchased from Tacara Sdn. Bhd. The fully hydrolyzed PVA used were Fully Hydrolyzed PVA with MW: 145,000 (Merck KGaA Darmstadt). Distilled water was used as the solvent.

B. Preparation of carrageenan solution

1 w/v % of CAR solution was prepared by dissolving 0.2 g of CAR in 20 mL of dissolved water at a temperature range of 80℃ to 90℃ [24]. The solution is then stirred with magnetic stirring until the solution was completely dissolved in order to obtain homogenous solution. The CAR solutions were left for an hour in order to eliminate any air bubbles that are formed in the solutions.

C. Preparation of polyvinyl alcohol solution

13 w/v % of fully hydrolyzed PVA solution was prepared by dissolving 2.6 g of fully hydrolyzed PVA in 20 mL of dissolved water at temperature ranging of 180℃ to 190℃. The solution is then stirred with magnetic stirring until the solution was completely dissolved in order to obtain homogenous solution. The PVA solutions were left for an hour in order to eliminate any air bubbles that are formed in the solutions.

D. Preparation of PVA/CRG solution

The PVA and CAR solutions were left to cool down before mixing. Then, the solutions were mixed using the magnetic stirrer with a ratio of 70:30 of the 13 w/v% PVA and 1 w/v% CAR solutions in order to be used as the polymer solution later in the electrospinning process.

E. Electrospinning

A 1 ml of PVA/CAR solution was transferred into a 5 ml syringe and loaded into the electrospinning apparatus. During the electrospinning process, high voltage power was applied to the PVA/CAR solution that was filled inside a syringe via an alligator clip that were attached at the tip of the syringe needle. The applied voltage was varied to be 9kV, 12kV, 15kV, and 18kV. The solution was then distributed to the blunt needle via a syringe pump in order to control the solution flow rate which were adjusted at 0.2ml/h, 0.3ml/h, 0.4ml/h, and 0.5ml/h. The electrospun nanofibers were collected at an aluminium foil collector that were placed 12cm, 15cm, 18cm, and 15cm from the needle tip shown in **Figure 1**. The collected nanofibers were then kept and maintained at an ambient condition for further characterization.

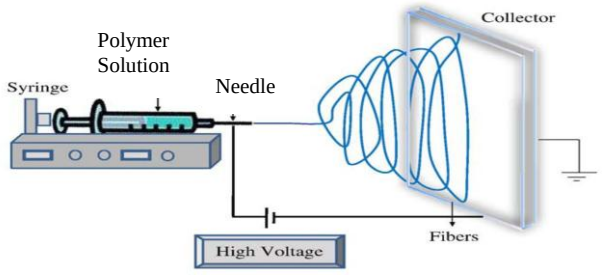


Figure 1: Schematic representation of the electrospinning setup.

F. Characterization

i) Scanning Electron Microscope, SEM

The morphology analysis for the nanofibers were observed using the Benchtop Scanning Electron Microscope (SEM), Hitachi TM 3030 Plus. Three (3) different magnification was used to obtain the clear image of the electrospun nanofibers which are 1kx, 5kx, and 10kx. 20kx magnification was used in order to measure nanofibers diameter.

ii) ImageJ Software

ImageJ software was used to determine the particle size distribution. 20kx magnification was used in order to measure nanofibers diameter using ImageJ software. At least 50 readings were used to determine the average mean fiber diameter.

iii) Contact Angle, CA

The equipment that was used to analysis the contact angle of the nanofibers is the VCA 3000 Water Surface Analysis System by AST Products Inc. the wetting properties were observed and therefore determining its absorption capabilities of the nanofiber surface. Water drop was fixed at 5μL to ensure uniform distribution. Time taken and image of initial liquid drop and final steady liquid drop condition was observed for analysis.

III. RESULTS AND DISCUSSION

A. The effects of applied voltage

The effect of applied current to the polymer solution through the needle of the syringe will cause the spherical droplet of the polymer solution to be deformed as Taylor cone and forming ultrafine nanofibers at a critical voltage (Haider et al., 2018). A series of experiment was conducted where the applied voltage was varied to 9kV, 12kV, 15kV, and 18kV. The distance between the tip of the needle to the collector was held at 15cm with flowrate 0.2ml/h. Results were shown in Figure 4.1 and Figure 4.3. The mean fiber diameter for flow rate shown on Figure 4.2. The estimated mean fiber diameters are 160.94 nm, 137.95 nm, 128.18 nm, and 119.69 nm in accordance with applied voltages 9kV, 12kV, 15kV and 18kV. From the SEM images, there are beads formation on the nanofibers with voltage 15kV and 18kV while smooth nanofibers observed with voltage 9kV to 12kV. From Figure 4.3, the contact angle analysis shows that each electrospun nanofibers with applied voltages 9kV, 12kV, 15kV and 18kV have decreased angle of 19.6°, 25.7°, 43.7° and 12.3° respectively, after the steady liquid drop. This shows that the nanofibers are hydrophilic [26].

An increase voltage enhance the electrostatic force to the solution causing it to form stretching of jet and thus forms to thin nanofibers (Someswararao et al, 2018). This is also in agreement of the study made by Lasprilla-Botero et al. (2018). They stated in their research that when the applied electric field is increased during the electrospinning process, the charged jet would move faster to the collector, having the solvent to evaporate in time and thus produces fibers with smaller diameter. A decreased mean fiber diameter can be noticed in Figure 4.1 from 160.94 nm to 119.69 nm. Optimum voltage is required in order to obtain thin nanofibers whereas higher voltage may lead to bead formation [27].

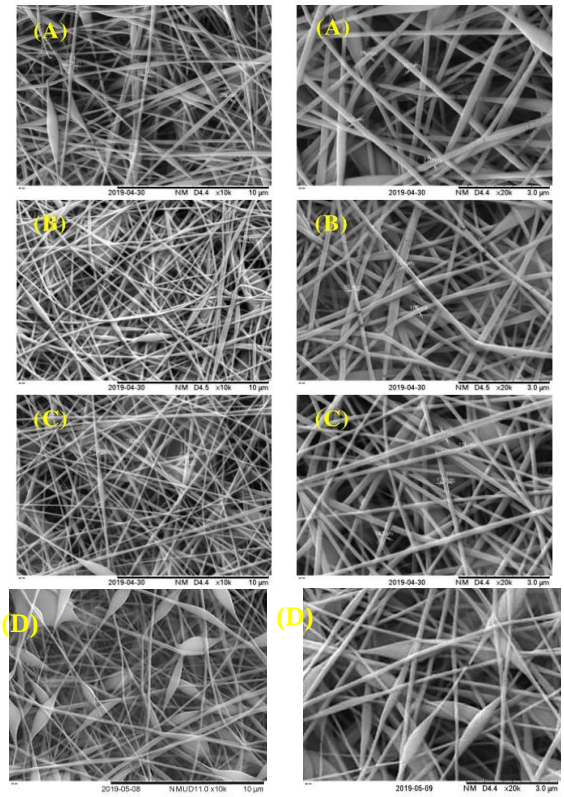


Figure 2: SEM images on effect of applied voltage on morphology of nanofibers (flow rate = 0.2 ml/h; distance = 15cm). Voltage: (A) 9kV; (B) 12kV; (C) 15kV; (D) 18kV. Original magnification of 10kx and 20kx.

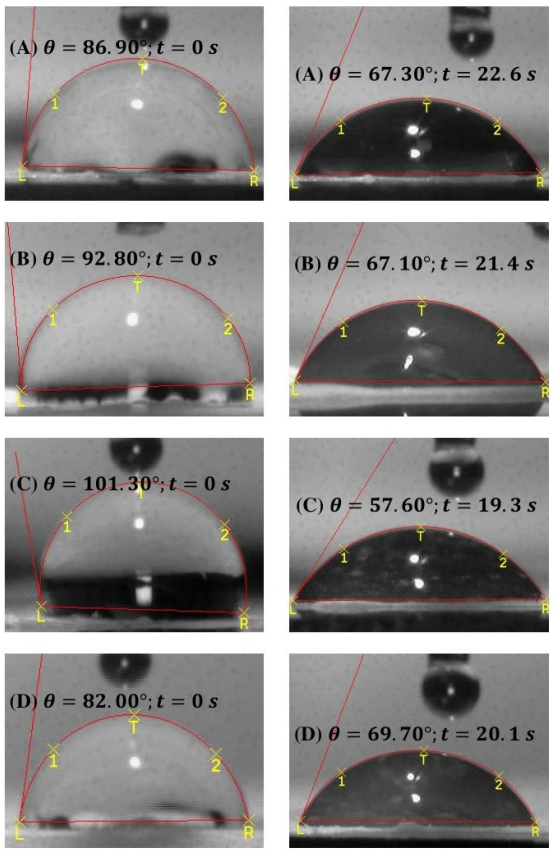
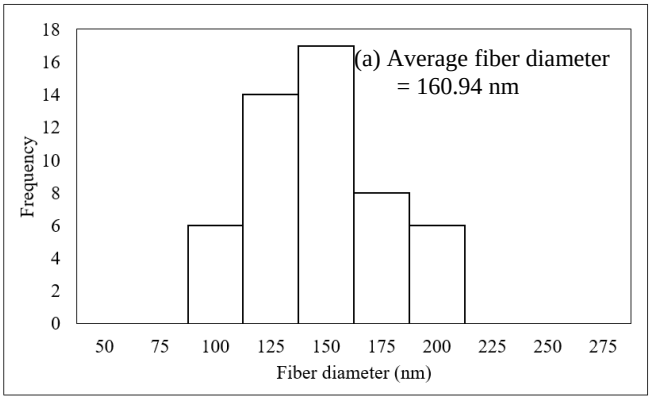
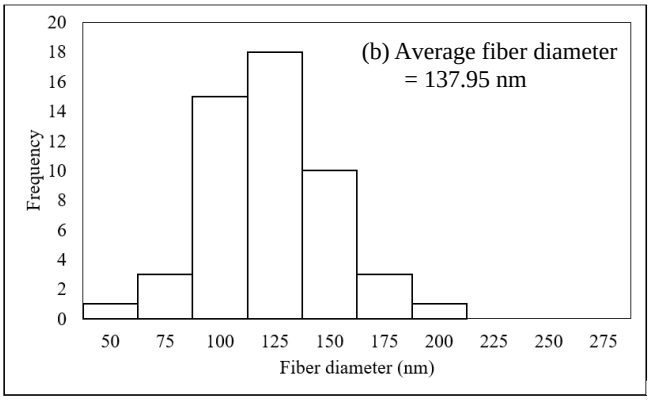
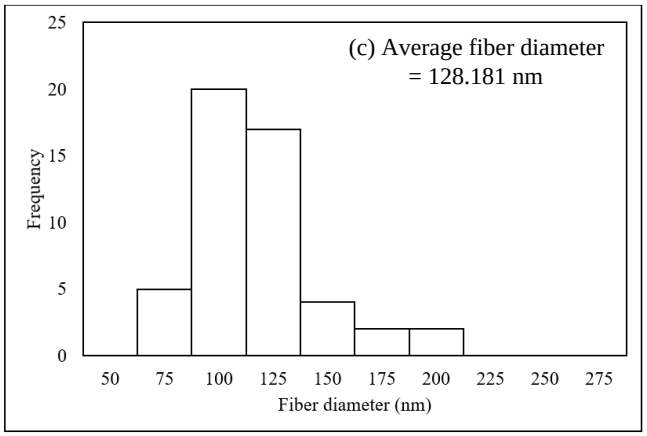


Figure 3: Contact angle on effect of applied voltage on morphology of nanofibers (flow rate = 0.2 ml/h; distance = 15cm). Voltage: (A) 9kV; (B) 12kV; (C) 15kV; (D) 18kV.







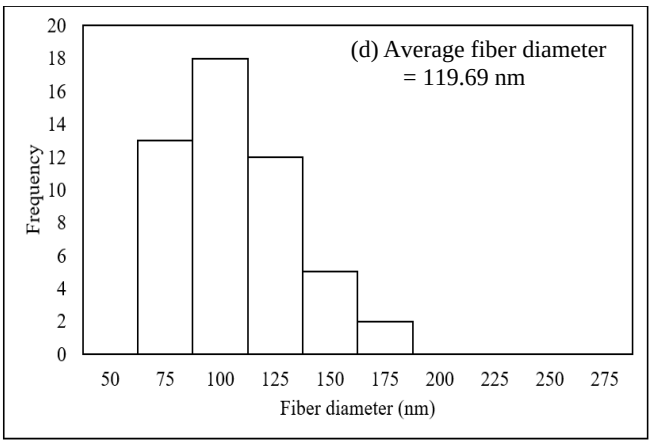


Figure 4: Particle size distribution histogram. Voltage: (A) 9kV; (B) 12kV; (C) 15kV; (D) 18kV. Average fiber diameter: (A) 160.94 nm; (B) 137.95 nm; (C) 128.181 nm; (D) 119.69 nm.

B. The effect of flow rate

Changes in flow rate of solution in the electrospinning process shown in Figure 4.4 and Figure 4.6. A series of experiment was conducted where the flow rate was varied to 0.2 ml/h, 0.3 ml/h, 0.4 ml/h, and 0.5 ml/h. During this process, the applied voltage and tip to collector distance were maintained at 12kV and 15cm respectively. As shown in Figure 4.5, it is observed that the mean fiber diameter is 129.52 nm, 133.85 nm, 138.73 nm, and 140.49 nm, in accordance with the flow rates 0.2 ml/h, 0.3 ml/h, 0.4 ml/h, and 0.5 ml/h. The contact angle analysis in Figure 4.6 shows that the electrospun nanofibers with varied flowrate of 0.2 ml/h, 0.3 ml/h, 0.4 ml/h, and 0.5 ml/h have decreased angle of 38.6°, 28.4°, 27.6° and 33.0° respectively, after the steady liquid drop. This shows that the nanofibers are hydrophilic [26].

Enough time is needed in order for the polymer solution to evaporate which can be obtained by selecting a smaller flow rate. Higher flow rate, however, would yields beaded fibers as there is lesser time for the evaporation of solution. From Figure 4.4, it is shown that there is a very significant formation of beads on the nanofibers in the films can be seen with flowrate of 0.4 ml/h and 0.5 ml/h. the high flow rate will the decreasing of charge density and thus increases diameter of fibers. Figure 4.6 shows the increment of nanofiber diameter from 129.52 nm to 140.49 nm. This is also in agreement with the finding made by Motamedi et al. (2017). They stated that in their study of electrospun polyvinylidine fluoride fiber shows that the increase of flow rate would resulted in a decrease of fiber diameter. Therefore, an optimal flow rate is preferred for producing fine and smooth nanofibers. [27].

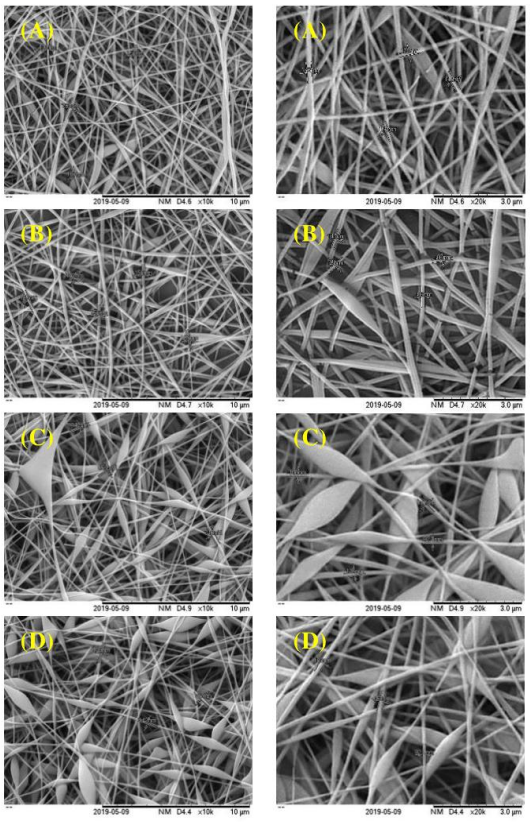


Figure 5: SEM on effect of flow rate on the morphology of nanofibers (voltage = 12kV; distance = 15cm). Flow rate: (A) 0.2 ml/h; (B) 0.3ml/h; (C) 0.4ml/h; (D) 0.5ml/h. Original magnification of 10kx and 20kx.

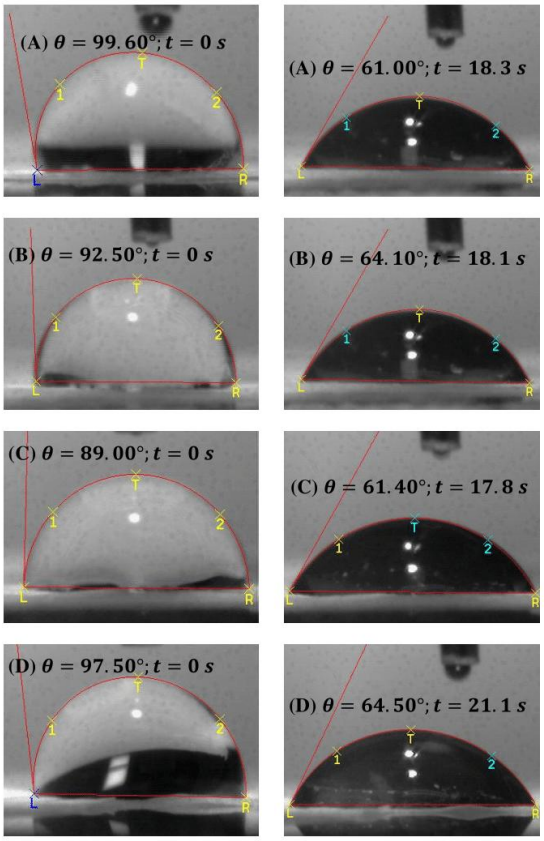
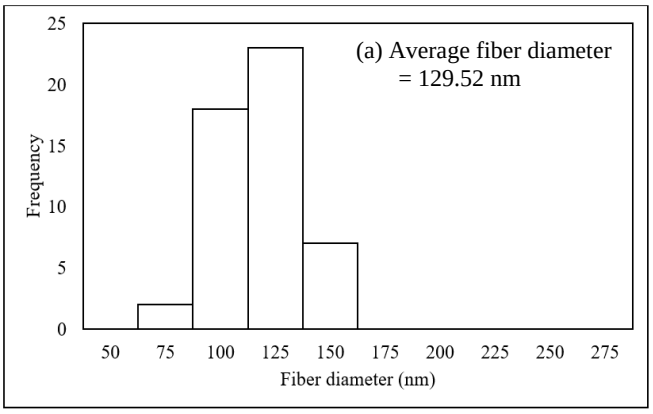
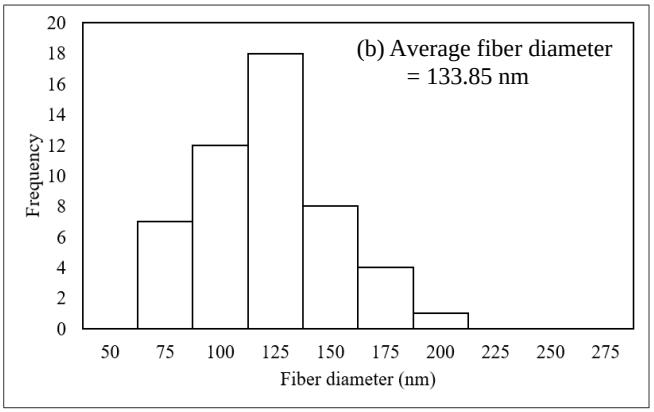
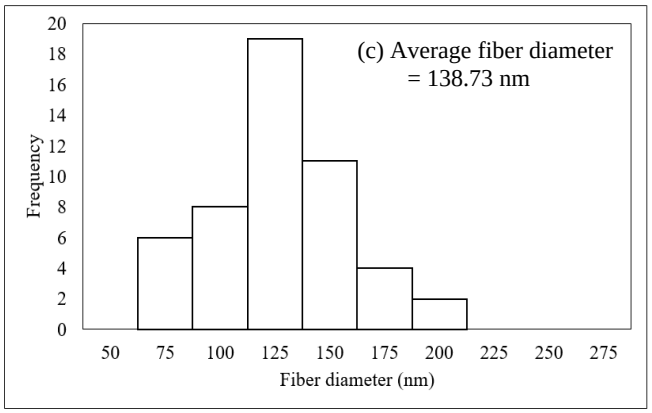


Figure 6: Contact angle on effect of flow rate on the morphology of nanofibers (voltage = 12kV; distance = 15cm). Flow rate: (A) 0.2 ml/h; (B) 0.3ml/h; (C) 0.4ml/h; (D) 0.5ml/h.







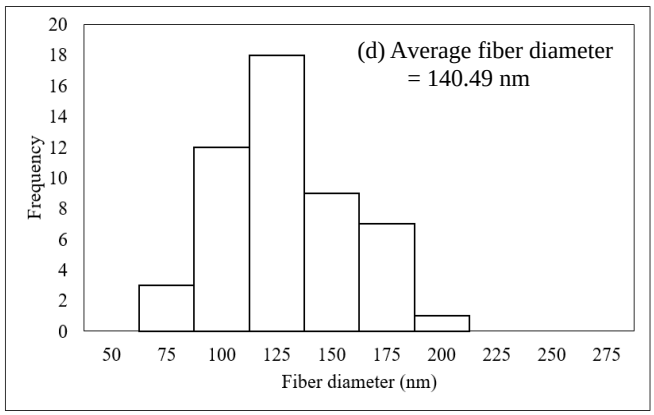


Figure 7: Particle size distribution histogram. Flow rate: (A) 0.2 ml/h; (B) 0.3ml/h; (C) 0.4ml/h; (D) 0.5ml/h. Average fiber diameter: (A) 129.52 nm; (B) 133.85 nm; (C) 138.73 nm; (D) 140.49 nm.

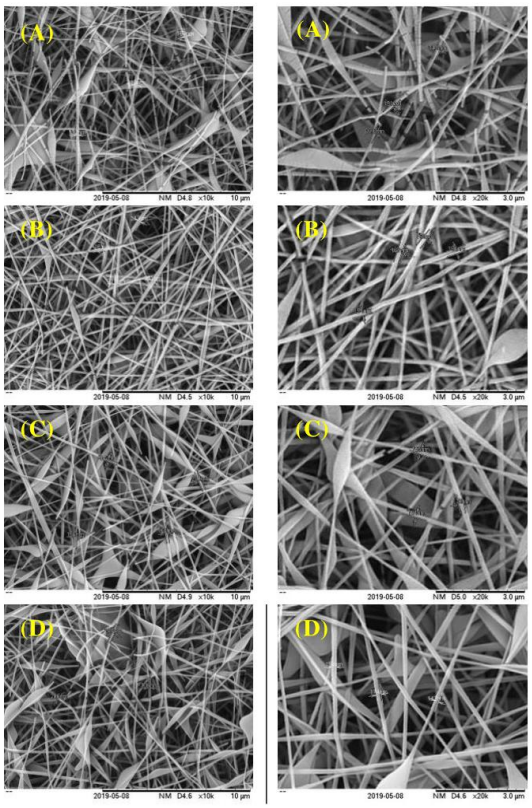


Figure 8: SEM on effect of needle tip to collector on the morphology of nanofibers (voltage = 12kV; flow rate = 0.2ml/h). Distance: (A) 12cm; (B) 15cm; (C) 18cm; (D) 21cm. Original magnification of 10kx and 20kx.

C. The effect needle to collector distance

The morphology of the electrospun PVA/CAR nanofibers prepared at different tip-collector distances 12cm, 15cm, 18cm, and 21cm shown at Figure 4.7. The applied voltage and flow rate was kept at 12 kV and 0.2 ml/h respectively. The mean fiber diameter calculated shown in Figure 4.8 are 132.94 nm, 131.79 nm, 124.38 nm, and 114.72 nm, corresponding to the tip-collector distance of 12cm, 15cm, 18cm, and 21cm respectively. Contact angle that were analyzed in Figure 4.9 shows that each nanofibers have a decreased angle of 38.4°, 36.8°, 18.7°, and 30.6°, after the steady liquid drop. This shows that the nanofibers are hydrophilic [26].

From the decrease mean fiber diameter from 132.94 nm to 114.72 nm, it can be concluded that increase distance tip to collector yields thin nanofibers. According to the study made Nurwaha et al. (2013), they analyzed that the fiber diameter decreases as well as increases by increasing of distance to collector. For a short distance, the solvent may not be able to fully evaporate due to not having enough time before the charged jet arrived at the collector. Increasing the distance to collector however results in decrease of fiber diameter since more time is given for the solvent to evaporate and the jet to stretch before reaching the collector. They also mentioned that at long distance, the electric field will decrease and this will affect the electrostatic field to be weaker. Thus, making the stretching of the charged jet to decreased and therefore increase fiber diameter. This can be concluded that the distance to collector is strongly influenced by applied voltage. Tip to collector distance is a parameter that affects the evaporation time required by the solvent where faster evaporation of the solvent would fabricate thinner fibers [27].

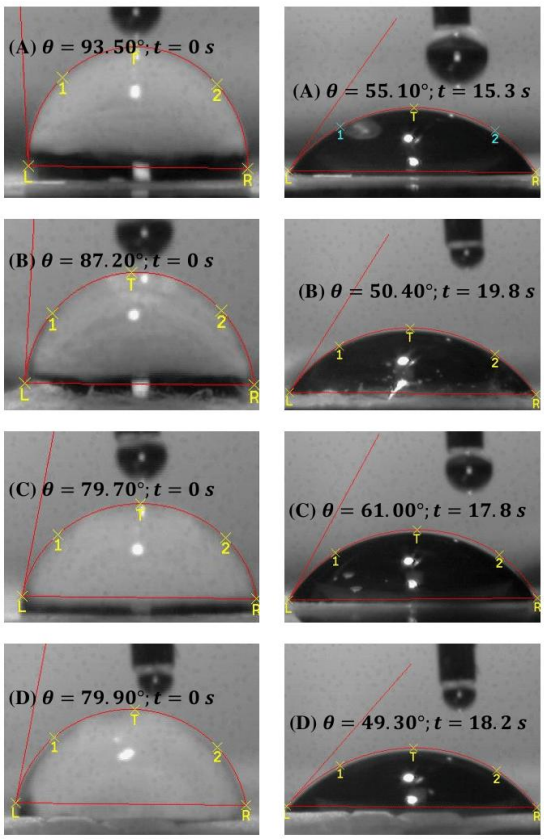


Figure 9: Contact angle on effect of needle tip to collector on the morphology of nanofibers (voltage = 12kV; flow rate = 0.2ml/h). Distance: (A) 12cm; (B) 15cm; (C) 18cm; (D) 21cm.

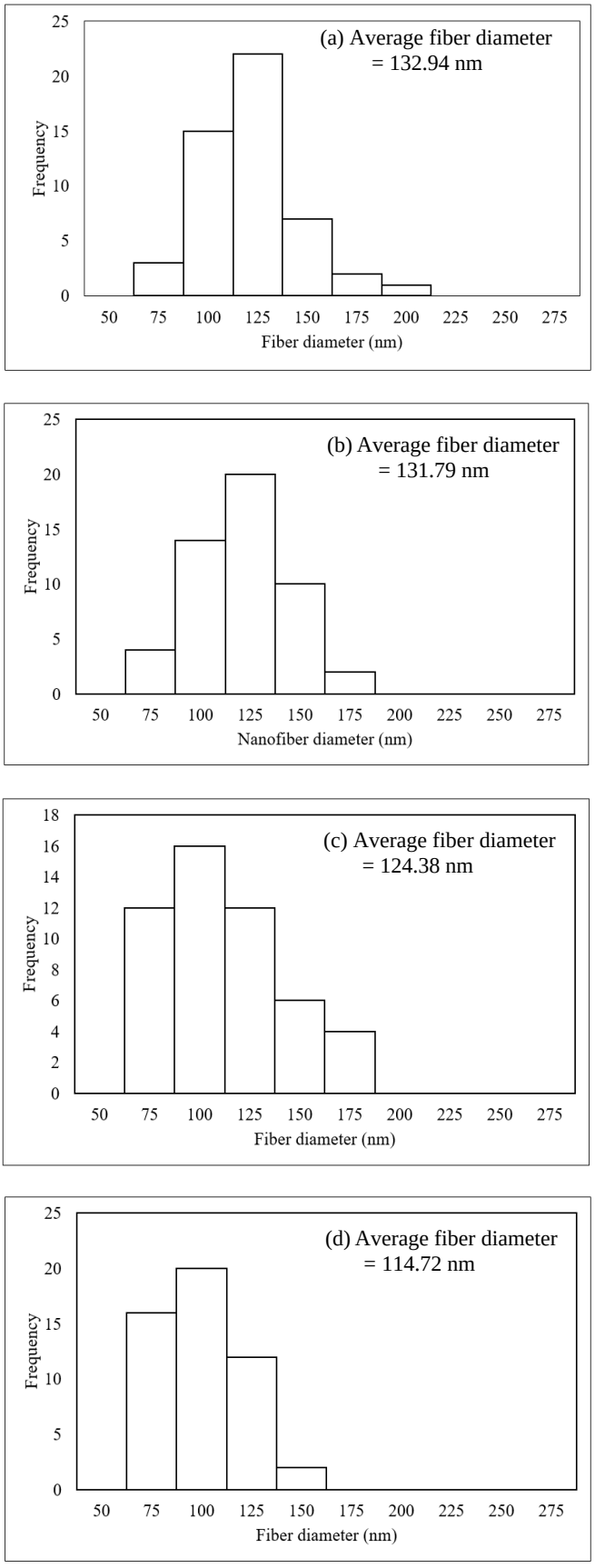


Figure 10: Particle size distribution histogram. Distance: (A) 12cm; (B) 15cm; (C) 18cm; (D) 21cm. Average fiber diameter: (A) 132.94 nm; (B) 131.79 nm; (C) 124.38 nm; (D) 114.72 nm.

IV. CONCLUSION

By using the SEM image analysis, the diameter of the nanofibers could be observed. ImageJ software enables calculation of the mean fiber diameter of the electrospun nanofibers image by SEM magnification of 20kx. From the study, the effect of increasing applied voltage of 9kV, 12kV, 15kV and 18kV results in decreasing fiber diameter from 160.94 nm, 137.95 nm, 128.18 nm, to 119.69 nm. By increasing the flow rates of 0.2 ml/h, 0.3ml/h, 0.4 ml/h, to 0.5 ml/h during the electrospinning process, the diameter of the nanofibers would increase from 129.52 nm, 133.85 nm, 138.73 nm, to 140.49 nm. The effect of increasing distance to collector shows yields a decrease in fiber diameter. The mean fiber diameter data collected was 132.94 nm, 131.79 nm, 124.38 nm, and 114.72 nm corresponding to the tip-collector distance of 12cm, 15cm, 18cm, and 21cm respectively. Contact angle shows that the nanofibers are water soluble and hydrophilic. Is can be concluded that optimum value of voltage, flow rate, and distance is required in order to fabricate fine and smooth nanofibers. This study is helpful to optimize the electrospinning process parameters for the preparation of PVA/CAR nanofibers whether according to desired mean diameter.

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