Chapter

Fabrication of PVA/Carbon-Based Nanofibers Using Electrospinning

Gatut Yudoyono, Diky Anggoro, Lutfi Fitria Ningsih and Rizki Romadoni

Abstract

Nanofibers are widely used in various fields, including water filtration. In the development of nanofibers as water filtration, a mixture of carbon in a polymer solution is often used. Nanofibers can be made by several methods such as multicomponent fiber spinning techniques, melt blowing, electrospinning. Electrospinning is currently a simple development method but can produce nanofibers with a small fiber diameter, it is easy to develop and many parameters can be controlled. Parameters that affect the results of the nanofibers that are formed include flow rate or syringe pump flow rate and high voltage dc high voltage. Various types of nanofibers can be produced from various types of polymers, both natural polymers and synthetic polymers. Generally, because they have properties and characteristics such as high surface area, small pore size, and the possibility to be developed in various applications. Therefore, this chapter discusses the electrospinning of carbon nanofibers using PVA polymer.

Keywords: DC high voltage, electrospinning, nanofibers, PVA/carbon

1. Introduction

Nanofibers are one of the nanotechnology products. Nanofibers are defined as an ultrafine dense fiber having a very small diameter. Nanofibers diameter is tens to hundreds of nanometers, so it is called ultrafine solid fiber (Nanofibers is defined as a fiber with a diameter of 100–500 nm [1]. Research related to nanofibers continues to be carried out for one of the reasons is, has the advantage of a large surface area per unit mass and small pore size and has superior mechanical properties [2], but the quality of nanofibers does not only depend on the chemical properties of the solution but rather the size and mechanical properties also very important [1, 3, 4]. Nanofibers size, density, mechanical properties, and orientation are essential for a reliable product application. The properties and characteristics of the fibers will change drastically when their size subside from micrometers to nanometers, one of which is the increase in the surface area of the fiber to its volume ratio, examples of these mechanical properties are stiffness and strength. With these best characteristics, nanofibers have a very extensive application [5], and nanofibers applications are widely used in various industries. Making nanofibers can be done by several methods, is a multicomponent spinning technique, melt blowing, and electrospinning [6, 7].

The type of nanofibers that is currently developing very rapidly in the field of research, material synthesis systems and product applications is carbon nanofibers (CNF). Carbon nanofibers (CNF) has applications as a promising material and has great potential in various fields, in the chemical field, carbon nanofibers (CNF) have been widely applied to gas and water membranes, by utilizing the advantages of CNF in porosity, surface area high as well as good higher chemical resistance [8]. In the field of physics, the good thermal and electrical conductivity properties make CNF very potential to be applied to electrical devices, batteries in the electrode material, energy storage and as a sensor [9]. While in the field of materials science, CNF has been applied to the strengthening of composites and supercapacitor materials [8, 10].

The precursor for forming carbon nanofibers consists of Polyvinyl Alcohol (PVA) and Carbon, PVA is a polymer that has flexible properties, can form hydrogel bonds, is easily broken down naturally and is often used in the formation of nanofibers [11]. The chemical structure of PVA is shown in **Figure 1**, the degree of hydrolysis of PVA is around 98.5% so that it can dissolve in water with a temperature of 70 ° C [12]. In addition, PVA has optical properties, a quite good load storage capacity but poor conductivity values. Therefore, to overcome the bad conductivity properties can be done by means of doping. The nature of PVA is colorless, odourless, tasteless, and soluble in water [13].

Because PVA has biodegradable properties, this is what makes this polymer widely used for its applications in the medical, food industry and electronics. The physical properties of PVA are presented in **Table 1** below [6]. Anita and Harsojo, in their research, explained that the morphological results of PVA fabrication using electrospinning owned by nanofibers which were formed at a concentration of 10% were continuous [15, 16].

Carbon is a material that has various advantages in terms of physical and chemical properties, so many researchers have developed it today. This advantage of carbon makes it a material with the extensive application. The performance of this carbon is influenced by morphology. This morphological difference will result in the wide application of the carbon, such as catalyst supports, adsorbents, gas storage, separation technology, battery electrodes, porous template materials, fuel cells, and biological cells. In addition, several carbon particles with certain morphologies will have different applications [17], besides carbon material is also an amorphous

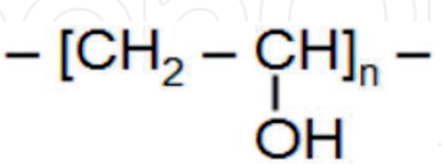

Figure 1.
PVA Chemical Structure.

Character	Value
Density	(1.19–1.31) gr/cm^3
Melting point	180–240 °C
Boiling point	228 °C
Decomposition temp	180 °C

Table 1.
Physical Properties of PVA [14].

compound which is produced from materials containing carbon or charcoal which are specially treated to obtain high adsorption power. Carbon can adsorb certain gases and chemical compounds, or its adsorption properties are selective, depending on the size or volume of pores and surface area. The absorption capacity of activated carbon is very large, namely 25–100% by weight of activated carbon [18].

The electrospinning method is a method that provides many advantages among the existing methods. This advantage is that the electrospinning technique can produce nano-sized fibers. The formation of jet polymer in the electrospinning method affects the morphological shape of the nanofibers, the polymer jet is influenced by environmental conditions, one of which is humidity [19], but most of the existing studies do not consider the relative humidity of the electrospinning spinning environment. Apart from relative humidity parameters, collector rotational speed affects fiber morphology. Collector rotating speed will affect fiber continuity.

2. Synthesis of carbon nanofibers

This section discusses the process of obtaining carbon nanofibers or the synthesis process to obtain carbon nanofibers which can be done, such as electrospinning (plate and drum collector), drawing methods and template methods. Each of these carbon nanofibers synthesis methods has its own advantages and disadvantages of the resulting material.

2.1 Preparation of carbon nanofibers (CNF)

The polymer solution in this study was made from polyvinyl alcohol (PVA), distilled water and carbon powder precursors with a size of 500 mesh. In the process of forming a polymer synthesis preparation material, PVA (molecular weight 60000, Merck Co) and carbon as a solute and distilled water as a solvent. The process scheme in making polymer solutions is by determining the concentration of the solution. The concentration of PVA solution that can be used in this study is 13 wt% with the solvent, and 2% wt carbon with distilled water as a solvent. After being measured, PVA and distilled water were mixed in one beaker. Then the magnetic stirrer is inserted into the reaction glass, then it is placed on the magnetic stirrer hotplate which has been activated. The temperature is set to reach 90 °C. After the temperature is right, this stirring process is carried out for one hour. Then the carbon and distilled water are mixed in one beaker glass, with the same steps as the PVA solution, the carbon and the solvent are placed on a hotplate magnetic stirrer which has been activated and the temperature is set to 30 After the temperature has been adjusted, the stirrer is turned on and the stirring process is carried out for one hour after the two solutions have dissolved well then, the PVA and carbon solutions are mixed with a volume ratio of PVA and carbon 2:1, then sonication is carried out by ultrasonic bath for 5 hours later. Stirred back at 30 °C for one hour.

2.2 Electrospinning

The electrospinning technique is a technology for making nano-sized fiber materials derived from materials in the form of solutions or liquids, as well as an efficient nanofibers manufacturing system by utilizing the influence of electrostatics in producing a solution (jet) of electrically charged polymer solutions or melts [20]. The electrostatic effect is generated by using a high voltage source. The voltage source that can be done in the use of electrospinning is between 7 kV to 32 kV [21]. Apart from the high voltage, the other important parts controlling the process are a

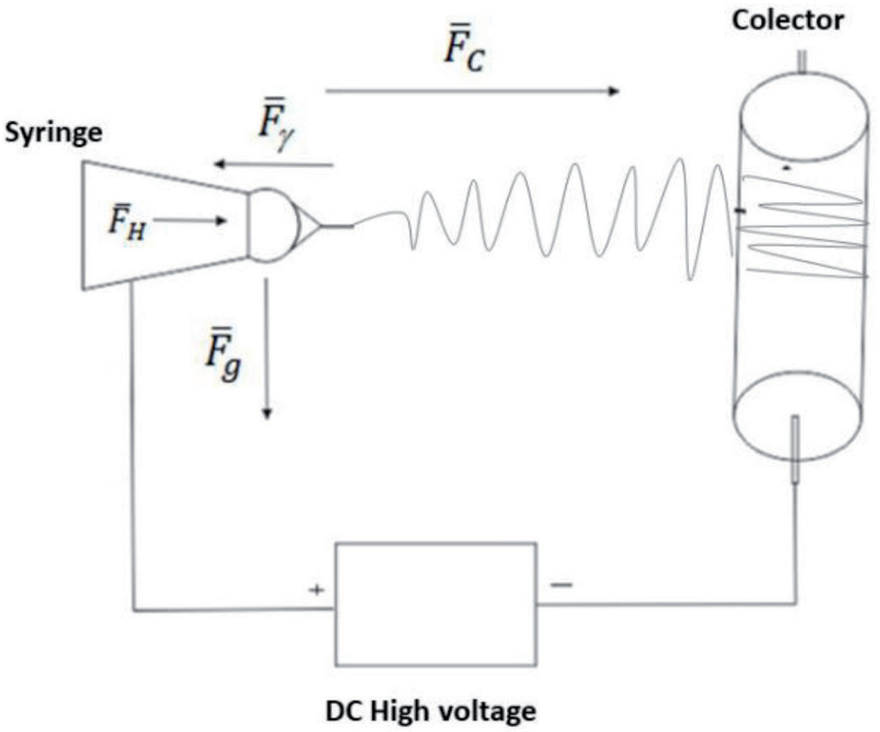

Figure 2.
The forces that appear in the electrospinning process.

syringe pump as a solution sprayer with a precise flow rate, and a collector as a place to collect the nanofibers that are formed [22]. When a high voltage is applied to the needle tip and collector, an electric field is formed around it. The positive pole is connected to the needle, the negative pole is connected to the collector. As the electric field around the needle increases, the hemisphere of the solution droplets at the tip of the needle will expand further and form a cone (also known as Taylor cone). When a high voltage is applied to the needle tip, the electric field will affect the surface tension of the solution droplet. Due to the influence of the electric charge on the needle tip on the solution, the solution is polarized and attracted towards the collector [23]. On the way from the tip of the needle to the collector, the solution undergoes thinning and evaporation of fibers or fibers that form and collect on the collector surface [21].

The forces acting on the electrospinning process can be described as in **Figure 2** which shows the modelling of the forces acting on the electrospinning process. From this figure, it can be written the equation of the forces acting on the electrospinning process as follows,

$$\bar{F}\gamma - \bar{F}_C + \bar{F}_H = 0 \tag{1}$$

when $\bar{F}\gamma$ is the surface tension force of the solution, $\bar{F}_C$ is the Coulomb force that arises because of the electric field, and $\bar{F}_H$ is the hydrodynamic force that occurs when the solution is pushed/pressed by the syringe pump [24].

2.3 Electrospinning with a plate collector

PVA and Carbon precursor solutions that have gone through the preparation stage will then be fabricated using an electrospinning system to produce carbon nanofibers, the electrospinning system used can be done with various schemes, but

the schemes used here use electrospinning with a stationary plate collector and a rolling drum collector.

As shown in **Figure 3**, is an electrospinning scheme with a syringe containing a polymer solution that includes a spinneret (needle), a direct current (DC) high-voltage power generator and a stationary collector plate. In the electrospinning method, a high voltage over a certain range is applied between two electrodes to obtain the desired type and quality of carbon nanofibers. The positive electrode is made in contact with the PVA + carbon fluid via a spinneret to produce a charged liquid when subjected to an external electric field, and the negative electrode is attached to a collector which acts as a fiber collector. Due to the electrostatic force, the solution will be attracted towards the collector.

Although such a simple plate-collector electrospinning scheme is sufficient to obtain fibers, it does not produce a homogeneous and evenly distributed CNF layer, as more CNF collects in the center of the collector, resulting in variations in thickness through the layers, which can also affect fiber morphology, as well as carbon nanofibers the resulting system tends to easily form beads as shown in

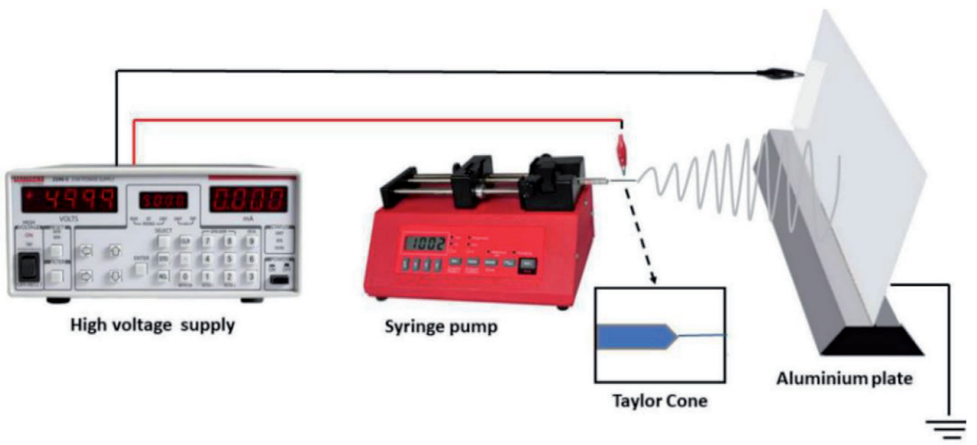

Figure 3.
Schematic illustration of an electrospinning system using a plate collector type.

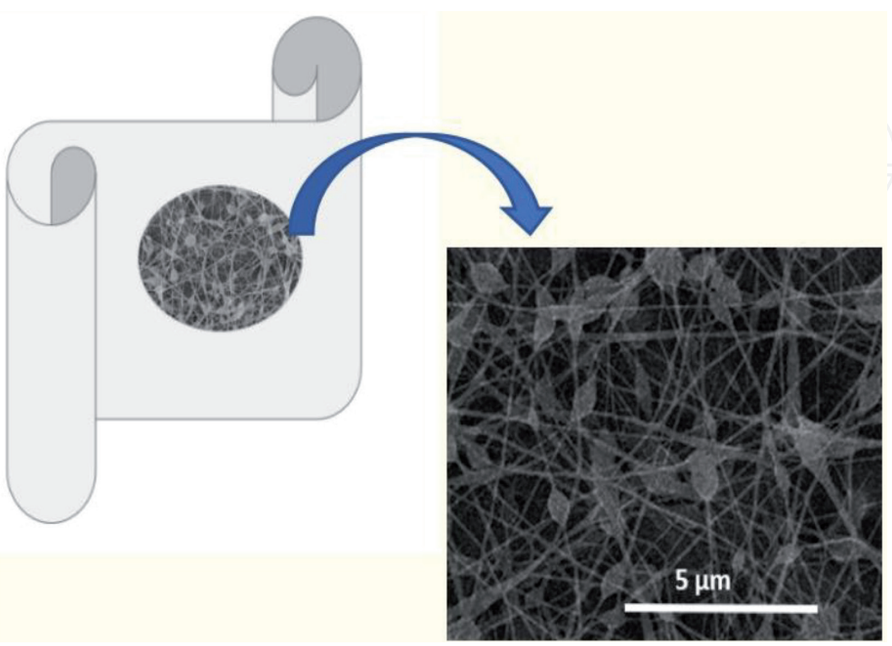

Figure 4.
SEM images of fabricated CNF using an electrospinning system with a stationary plate collector and most of the fibers are formed only in the center of the collector.

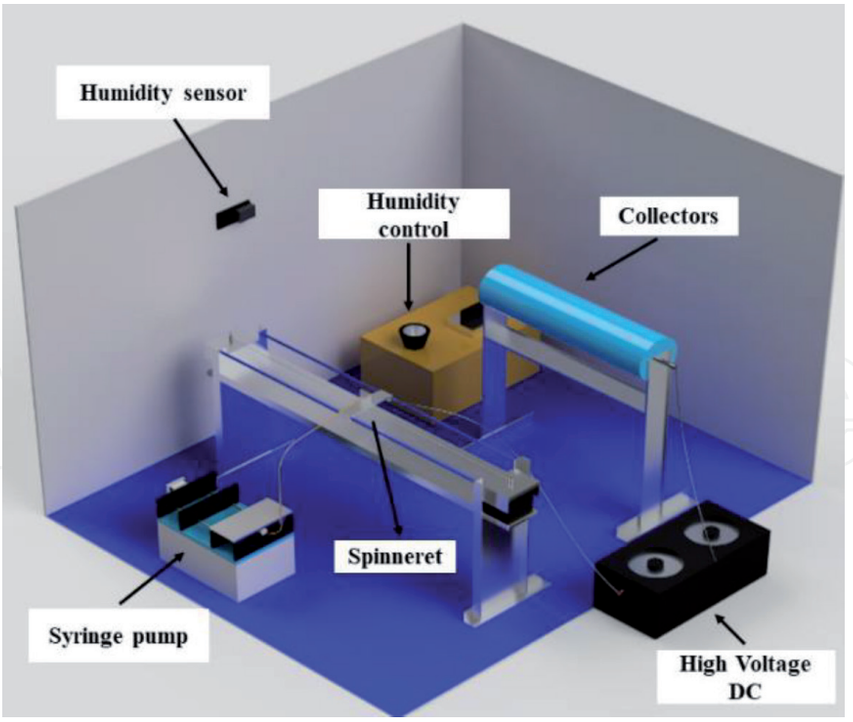

Figure 5.
Schematic illustration of an electrospinning system that uses a rotating drum collector type, with more complex controls and parameters.

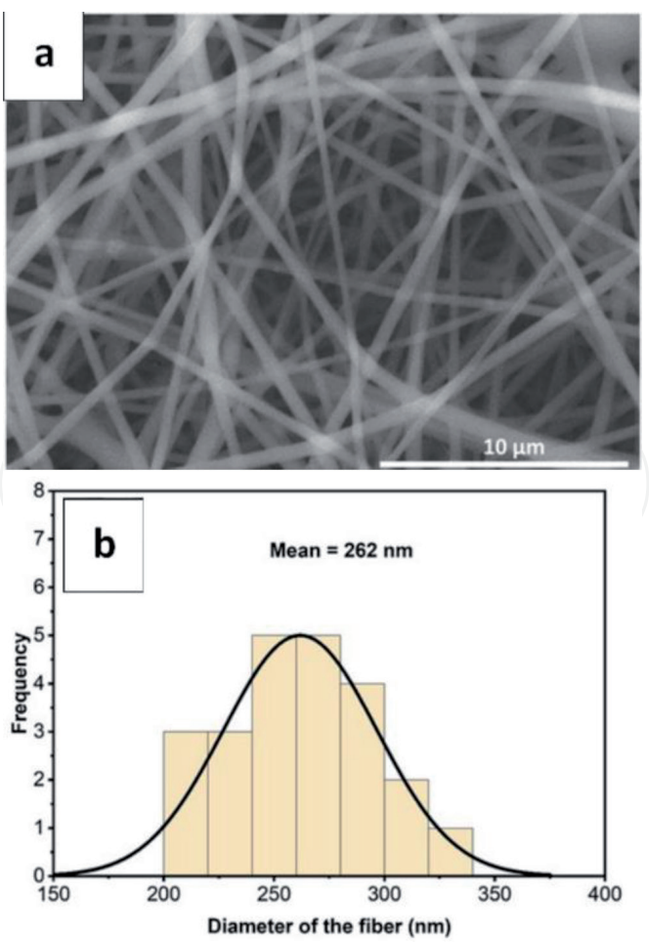

Figure 6.
(a) SEM image results, and (b) size distribution of CNFs from electrospinning fabrication results with rotating drum collectors, with a rotation speed of 130 rpm, a given high DC voltage of 10 kV and a relative humidity of about 30%.

Figure 4. The results were not homogeneous, and beads appeared on the CNF because the plate collector was used in a stationary or stationary position so that the effect of spinning fibers after the Taylor cone and jet polymer processes was very weak.

2.4 Electrospinning with a rolling drum collector

In this regard, electrospinning with a rotating drum collector has been developed to allow the formation of carbon nanofibers homogeneously and thoroughly to all areas on the drum surface, resulting in a CNF of uniform thickness as shown in **Figure 5.** When the drum collector rotates, the fibers will be attracted towards the collector and subjected to a spinning effect on the rotating drum, then the spinneret moves right and left, as well as the influence of the electric field between the needle and the collector which causes the CNF to form evenly throughout and formless beads in the layer collector.

Carbon nanofibers (CNF) formed with this system has an interesting material morphology as shown in **Figure 6** which is attractive in the sense that no beads are formed on the CNF, and the fibers are evenly distributed with a homogeneous thickness. The fiber that is formed enters the nanometer scale area, from direct measurements the diameter of the CNF formed is at 262 nm, as we all know that the limitations of fiber or composite materials are said to be in the nanoscale if they have a size of 50 nm to 500 nm.

3. Electrospinning control parameters

The electrospinning method has many parameters that must be controlled to produce nanofibers. The parameters that influence are high voltage, field, electricity, nozzle to collector distance, solution concentration, and humidity. The formation of jet polymer in the electrospinning method results in the morphological shape of the nanofibers. The polymer jet itself is influenced by environmental conditions, one of which is humidity. Humidity parameters greatly affect the diameter of the nanofibers, at high humidity, the fiber diameter will increase (Medeiros et al., 2018). The application of high voltage to electrospinning is very important in influencing the diameter and morphology of the nanofibers. The increase in high voltage causes an increase in the electric field as well as this affects the decrease in the diameter of the nanofibers and shortens the time of the solution from the tip of the needle to the collector. The flow rate in electrospinning is the flow of fluid from the syringe pump to the collector. The rate of solution (flowrate) affects the formation of fiber diameter and morphology. This process affects the material transfer rate and jet speed. The diameter of the fiber will increase as the rate of solution used increases [25].

Viscosity is the thickness of a solution; this viscosity is influenced by the concentration of the solution. High viscosity is difficult to force the solution out of the syringe so that the control on the needle is unstable, the higher the viscosity, the higher the fiber diameter. The diameter and morphology of the nanofibers are basically influenced by the distance between the needle tip and the collector. Distance affects fiber diameter and morphology because distance can determine the deposition time, evaporation rate, and polymer jet instability [26]. Therefore, an optimum nozzle to collector distance is needed to form carbon nanofibers with the desired diameter and fiber morphology. Several studies have studied the effect of the nozzle to collector distance and concluded that increasing the distance makes the fiber diameter decrease but the polymer jet instability increases [25].

The effect of relative humidity on the morphology of carbon nanofibers polymers is about the size of the diameter of the nanofibers which is strongly influenced by humidity during the spinning process seen from **Figure 7**. An environment with high humidity helps skin form rapidly with clear boundaries, whereas if there is less moisture, solvents will easily evaporate. When the polymer is hydrophobic, water acts as a non-solvent so that the fiber shell is more easily formed. This makes PMMA, PVC,

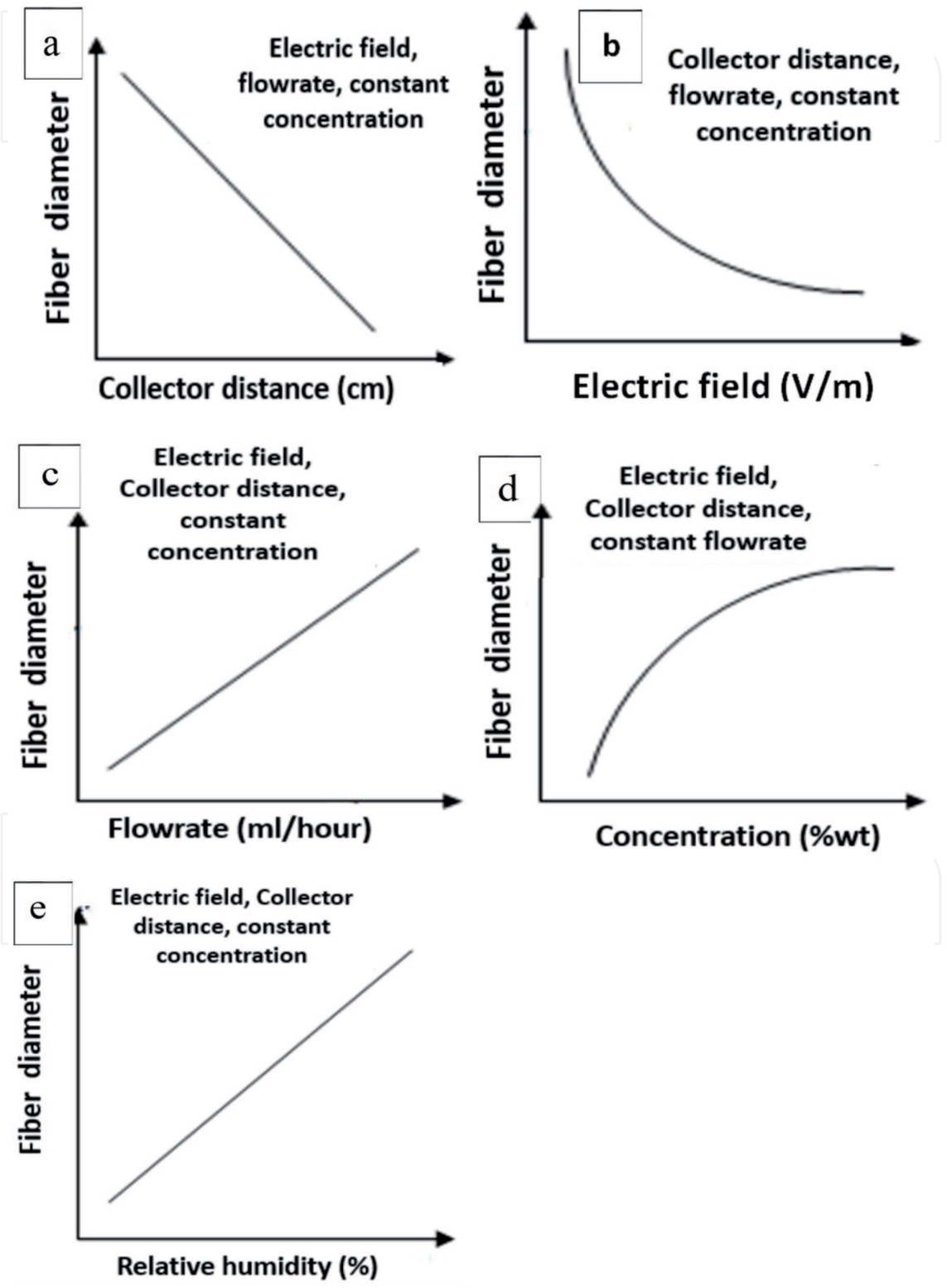

Figure 7.
Parameters affecting the size of the diameter of the nanofibers. (a) collector Distance (cm), (b) electric field (V/m), (c) flowrate (ml/hour), (d) concentration (%wt), (e) Relative humidity (%).

or PS in DMF, PMMA or PS in toluene have porous fibers when electros are in an environment with a relative humidity of more than 30%, while PVA is a hydrophilic polymer solution, water acts as a solvent so that the formation skin is easy to form humidity 20%. and there was no pore formation at all at a relative humidity value of 20% -80%. PVA nanofibers were successfully made in the relative humidity range of 20% -80%, but at high relative humidity, the morphology of the fibers contained more beads [27].

4. Characterization of carbon nanofibers (CNF)

The characterization carried out on the CNF depends on the application to be performed on it, the CNF which has been fabricated is researched as a sensor and as a Capacitive Deionization (CDI) electrode. To find out and measure the morphology and diameter of the nanofibers, a Scanning Electron Microscope or SEM is used for short. Also from SEM we get EDX data which shows the number of elements present in the CNF sample. To determine the electrical properties of CNF, a four-point probe characterization method was used, namely by using the I-V meter four probes. The measurement results using the I-V meter show the

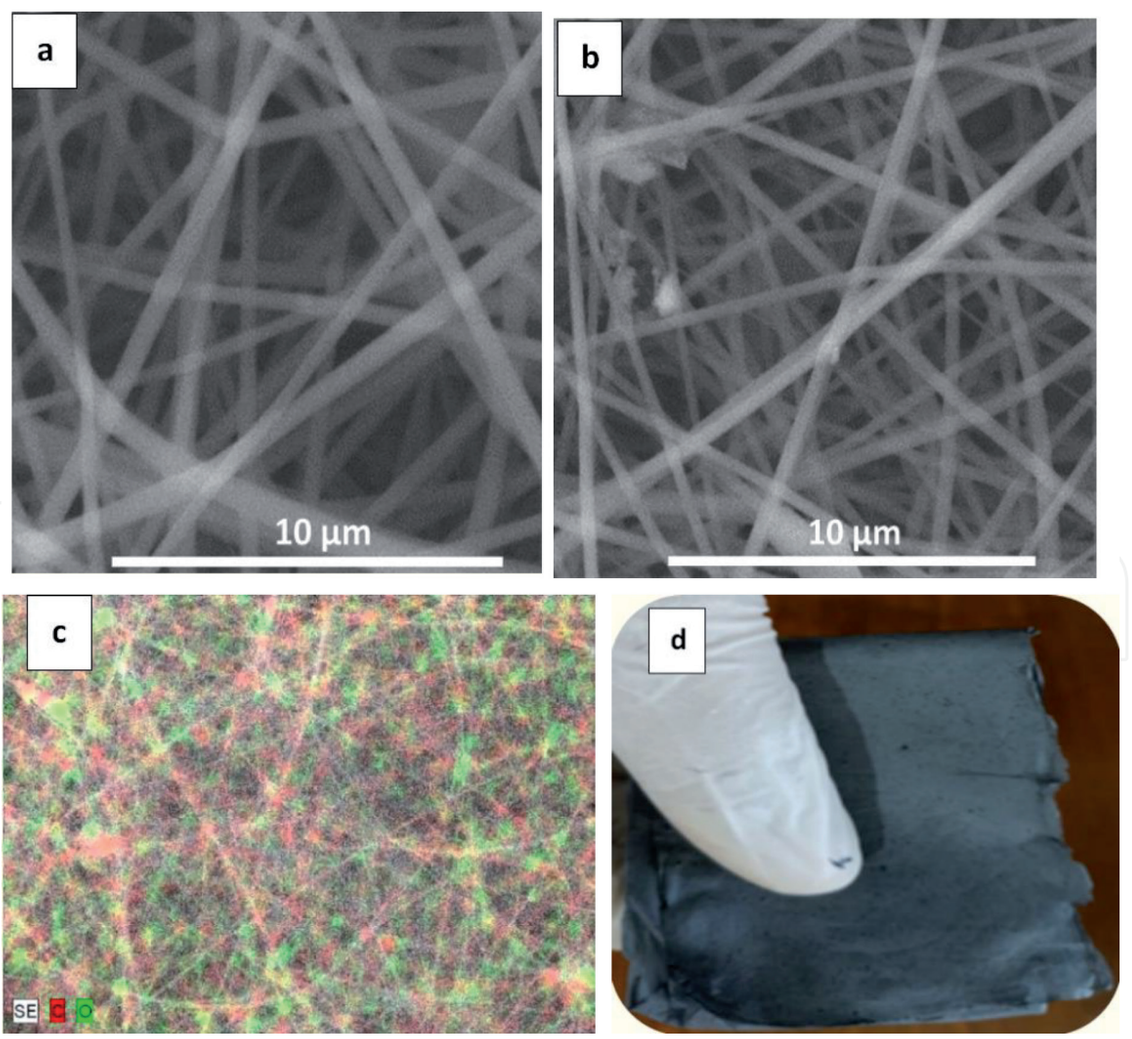

Figure 8.
Carbon nanofibers (CNF) electrospinning results with a flow rate of 0.1 ml/hour and relative humidity (a) 30% d and (b) 40% and an average fiber diameter of 262 nm (a) and 309 nm (b), (c) distribution of Carbon nanofibers elements at a flow rate of 0.5 ml/hour and relative humidity of 40% and (d) CNF sheets that are ready to be applied.

Ohmic curve on the I-V graph according to Ohm's law. The resulting Voltage and Current data are then processed according to Ohm's law to determine the conductivity value of the material.

Figure 8 shows the electrospinning results with a flow rate of 0.1 ml/hour and relative humidity of 30% and the parameters still produce fibers with an average diameter of 262 nm, the fibers in this parameter have a morphological shape with minimum and almost no beads. Whereas the electrospinning carbon nanofibers with a flow rate of 0.1 ml/hour and relative humidity of 40% and the parameters still produce a fiber with the smallest size of 200 nm and the largest diameter of 400 nm and an average diameter of 309 nm, the fiber in this parameter has a morphological shape with minimum and almost non-existent beads and more uniform diameter sizes and a narrower size range when compared to 30% humidity parameters. The CNF fabrication results were carried out by SEM–EDX to determine the

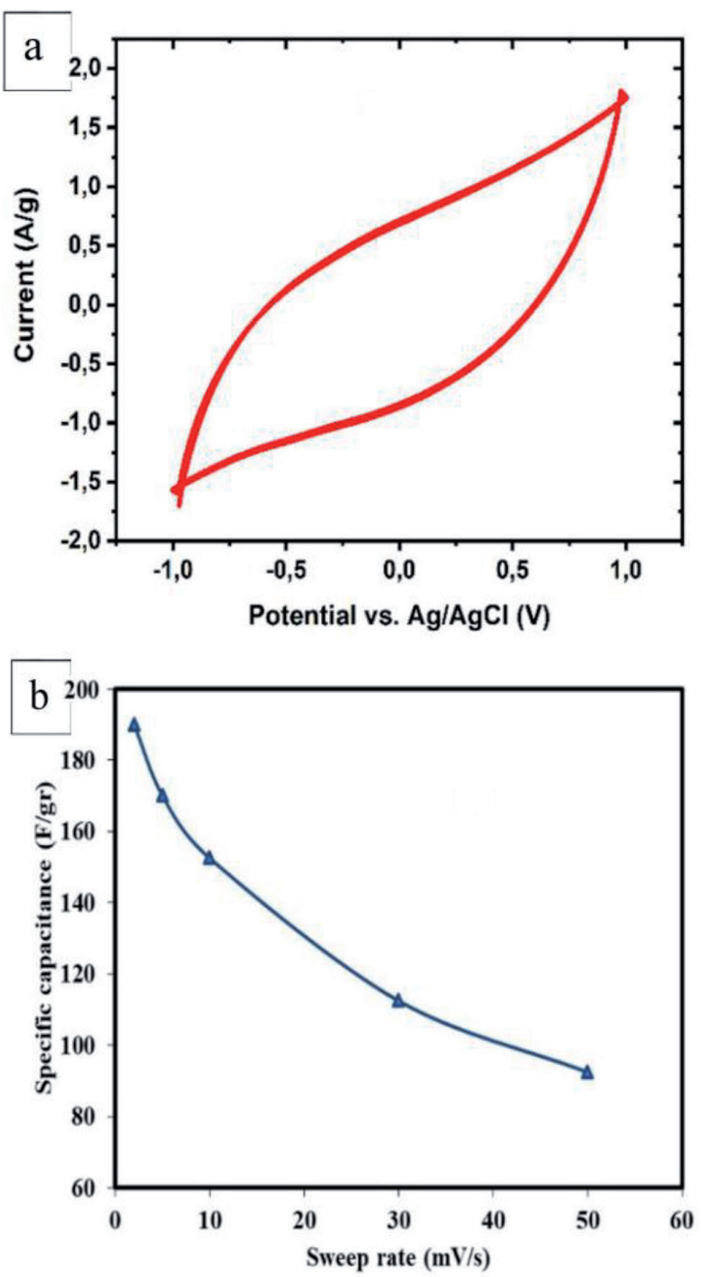

Figure 9.
Electrochemical characterization results. (a) diagram of the cyclic voltammogram (CV) data on the CDI electrode with a sweep rate of 5 mV/s, and (b) the specific capacitance of the carbon electrode from the cyclic voltammogram data.

Sample	CNF diameter (nm)	Voltage (mV)	Sheet resistance (Ω/sq)
A	417	28.7510	19.9822
B	358	26.8532	18.6633
C	309	26.5402	18.4457
D	262	25.6280	17.8117

Table 2.
The Average Value of Voltage and Resistance Sheet of Nanofibers from measurements using the I-V meter four probes.

elements of the nanofibers fabrication as in **Figure 8c**, the distribution of carbon elements has been evenly distributed, as well as the results of nanofibers fabrication with flow rate parameters of 0.1 ml/hour and 40% humidity and fixed parameters. Based on this characterization, carbon nanofibers consists of several chemical elements. The chemical elements, namely, O (Oxygen) by 24.57%, and C (Carbon) by 75.43%.

The cyclic voltammogram (CV) curve was obtained by scanning at a potential sweep rate of 5 mV/s on CNFS which had undergone temperature treatment and became a CDI electrode as shown in **Figure 9**. The CV curve shows the ideal behavior of the capacitor, the ideal CV curve with an almost square shape at different scanning sweep rates. Cyclic voltammetry testing on the three types of electrodes used in this study was carried out in a potential range of −0.5 V to 0.5 V and a potential sweep speed of 5 mV/s. The electrodes were immersed in an electrolyte solution of 0.5 M KCl with a submerged surface area of 1 cm2. Cyclic voltammetry measurement experiments were carried out at room temperature of 25 ° C. The results of the cyclic voltammetry test can be seen in **Figure 9**. On the voltammogram graph, a redox reaction is formed in an up-current pattern that shows the transfer of electrons from the electrode to the electrolyte solution, which involves the transfer of electrolyte ions based on the change in potential applied to the electrochemical cell, so this pattern increases in current indicating an increase in ion absorption capacity and ion absorption rate at potential given.

Based on **Table 2**, there is the largest sheet resistance value owned by CNF with the largest fiber diameter, but the difference is not too significant, the smallest sheet resistance value is owned by CNF with the smallest fiber diameter. The resistance value is directly proportional to the resistivity, but the resistivity is inversely proportional to the conductivity. Thus, the sample with the highest sheet resistance value has the greatest resistivity but the lowest conductivity.

5. Applications of carbon nanofibers

The application of CNF continues to develop today, based on the characteristics of CNF in the form of morphology and electrical properties, CNF has the potential to be applied to various fields, as shown in **Figure 10**, applications of CNF can be developed in the field of sensors, environmental applications and fields of electronics and electronics. Optical devices including energy storage fields.

Based on the characterization on the morphology and electrical properties of CNF, it is very possible to apply to sensor devices, the principle of chemiresistor sensors is more widely used for gas sensors because these sensors can be made easily and at relatively low cost. The chemiresistor mechanism is the reaction that occurs between the layers on the electrode and the gas which will result in a change in the value of resistance or conductivity.

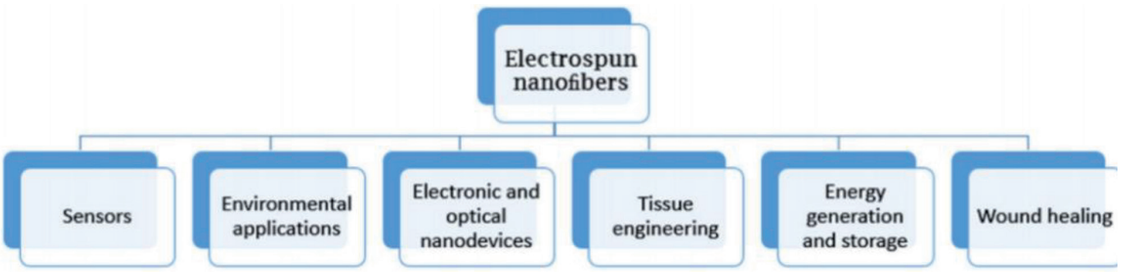

Figure 10.
General application areas of Carbon nanofibers [1].

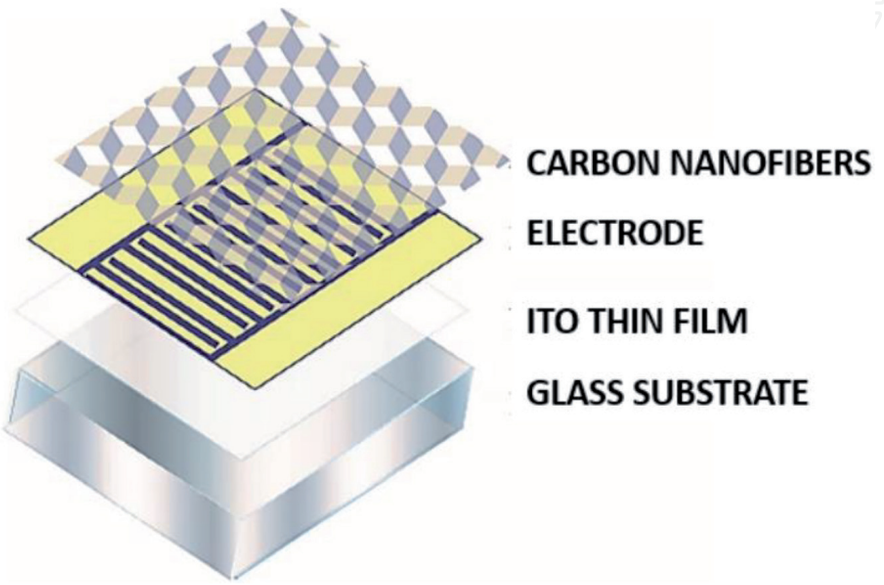

Figure 11.
Schematic of CNF application in sensor device fabrication.

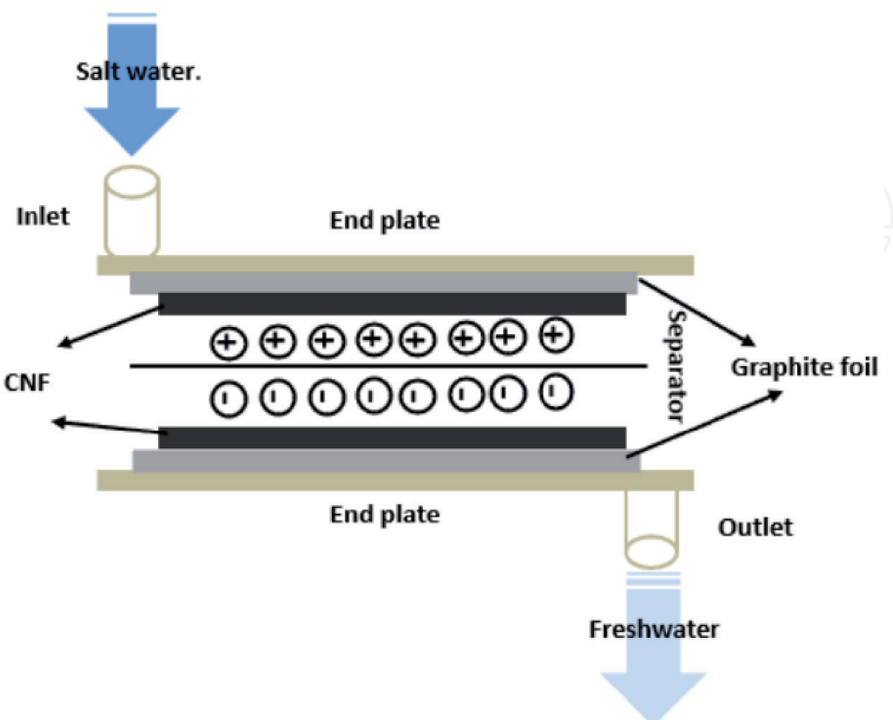

Figure 12.
Schematic of CNF application in electrode capacitive deionization CDI) [20].

This change occurs due to the transfer of valence electrons to the atoms of the sensor material due to the reaction with the reactant gas. The changes that occur are decreased resistance or increased conductance. Conductivity indicates the ability of a material to conduct electric current. The conductivity value can be determined by the equation below.

$$\sigma = \frac{1}{\rho} \tag{2}$$

Where σ is electrical conductivity (1/Ω.m) and ρ is electrical resistivity (Ω.m), the CNF application scheme for sensors can be seen in **Figure 11**. Whereas CNF which has been fabricated has high porosity and very high capacitance values. Good for capturing salt ions, it is very potential to be applied to Electrodes For Capacitive Deionization (CDI) with the scheme that can be seen in **Figure 12**.

6. Conclusions

Synthesis of CNF using electrospinning is an important part of this research, the synthesis process plays an important role in the quality of the CNF produced. In addition, the results of characterization of the different morphological structures of the CNFs formed show different properties, so that surface modification can help make CNF compatible for various applications. The characterization results show that the electrospinning result of the CNF composite diameter is in the range 200–450 nm, this shows that the electrospinning process with a rotating drum collector has advantages compared to a stationary plate type collector. Based on the electrical properties of the CNF I-V meter measurement results, a good conductivity value is obtained, which is due to the larger surface area of the CNF making it easier for electrons to move freely, so that CNF has the potential to be a good and modern sensor material. Whereas from the capacitive properties measured by the cyclic voltammogram (CV) curve with 5 mV/s and a measuring temperature of 25 °C shows the ideal behavior of the capacitor, the ideal CV curve with an almost square shape at different scanning sweep rates, so the use of CNF as a capacitive electrode is proper application.

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Conflict of interest

The authors declare no conflict of interest.

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