Effect of processing parameters on nanofibers via water vortex electrospinning

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Abstract—**Electrospinning is recognized to be flexible process which able to produce fibers made of any compositions with desired diameter from microns to several nanometers. Many methods have been developed for improvement and to overcome the limitations regarding the produced fibers. In this work, water vortex was used as one of the method to produce nanofibers in form of a yarn. The aim of this work was to produce nanofibers from water vortex electrospinning technique. The morphological structure for the produced fibers were characterized using scanning electron microscope (SEM). The structures of the produced fiber were analyzed.**

Keywords— Electrospinning, Nanofiber, Nanofiber yarn, Tissue scaffold, Water vortex

I. INTRODUCTION

The development of nanotechnology has given a huge advantage to many sectors such as energy, health, textile, defense system. Many of the sectors have incorporate the use of nanotechnology in their new development to produce a material, devices or system in nano size[1-3]. The novel chemical, physical and biological properties of nanomaterial size can be attributed to its unique shape and morphology to be applied in the industries. Tissue engineering is one of the example that has attract attention as a fusion technology that enable the regeneration of tissues and organs lost cause by disease or accident[4]. Even though there are many other methods to replace the lost of tissue such as autografts and allografts, there are still problem arise such as the availability of suitable harvest sites and rejection from mismatched donor – recipient pairs. The problem can be overcome by manufacturing of artificial porous structures called scaffolds[5]

Recently, the used of nanofiber as a base of scaffold systems are being explored for tissue engineering. The development of nanofibers has further improved the scope for scaffolds fabrication that have the potential to imitate the architecture of natural human tissues at nanometer scale. The characteristic of nanofiber such as high surface area to volume ratio, microporous structure that favors cell adhesion, proliferation, migration and differentiation which are highly desired properties for tissue engineering[6]. The conventional electrospinning method will produce fibers with randomly oriented structures in the form of nonwoven nanofiber mats or webs[7]. There is some limitation for the nonwoven structure. However, the used of nanofiber yarns have more great potential as it possesses much greater surface area and stiffness. Nanofiber yarns are also easier to handle instead of single nanofibers.

Electrospinning or electrostatic spinning is a simple technique which utilizes high electrostatic forces for fiber production. Electrospinning, first introduced by Formhals and later revived by Reneker uses high voltage (about 10–20 kV) to electrically charge the polymer solution for producing ultra-fine fibers (diameters ranging from a few nanometers to larger than 5 μm)[8]. In a typical process, an electrical potential is applied between a droplet of a polymer solution or melt, which is held at the end of a capillary tube, and a grounded target. To fabricate nanofibers, the collector is normally connected to a counter electrode, and the polymer solution is pumped through the needle of the syringe. The needle is in turn connected to a high-voltage power supplier, which supplies a voltage between 1 and 30 kV. In the presence of an electric field, the polymer solution at the tip of the needle becomes electrostatically charged, and forms a Taylor cone. The jet is accelerated by the electric field, and becomes thinner as it moves toward the grounded collector. Finally, the jet solidifies into a nanofiber[9, 10]. There are three critical parameters that will affect the characteristic of nanofiber which are the polymer solution, processing parameters and ambient conditions. In order to achieve desired fiber characteristics, the parameters can be manipulated[11-13].

The aim of this work was to produce nanofiber yarns from polyacrylonitrile (PAN) polymer using liquid medium. Water vortex was used to drawn down the nanofiber in a rotating manner that produces yarns. The process parameters such as applied voltage, solution flowrate and water vortex flow rate were manipulated in order to examine the morphological structure of the fibers. The nanofiber produced from water vortex was examined using scanning electron microscope (SEM). The characteristic of the produced fibers such as diameter was analyzed to see the variation with the manipulation of process parameters.

II. METHODOLOGY

A. Materials

Polyacrylonitrile (PAN) with Mw 100,000 g/mol was provided by Hochschule Hannover Mechanical Engineering Laboratory. Dimethylformamide (DMF) was used as a solvent in the experiment

B. Sample Preparation

A 20 wt% solution of PAN was prepared by dissolving PAN powder in DMF. The solution was stirred at 80 °C for 2 hours continuously using electromagnetic stirrer to get homogenous solution

C. Electrospinning Process

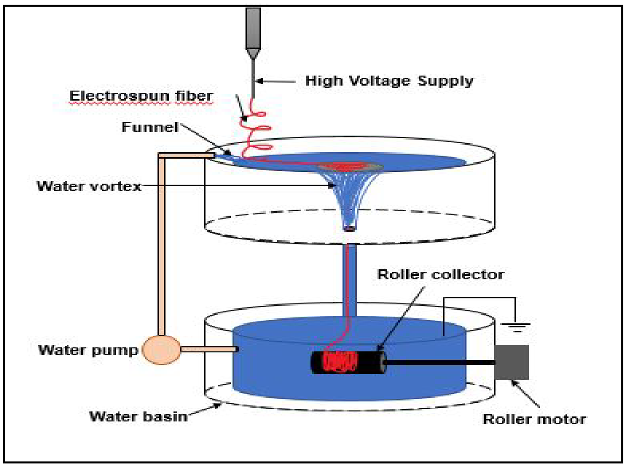

Fig. 1: Schematic set up for the production of nanofiber yarn by water vortex

Electrospinning process using water vortex was carried out to produce the nanofiber. Fig. 1 shows the schematic experimental set-up used to generate continuous nanofiber yarn. A cone shaped funnel was constructed to be a platform for the water vortex. The water vortex was formed on the funnel and drained out through a hole at the base of the funnel into a water basin placed below the funnel. A water pump was used to circulate the water from the water basin back to the funnel to produce a continuous water vortex and maintaining a constant water level in the basin. A grounded wire was placed inside the water basin to remove any residual charges on the water surface. A rotating collector moving by a roller motor was placed in the water in the water basin to wind up the fiber yarn. A grounded earth wire was inserted into the basin to remove any residual charges on the water surface. The electrospinning process was carried out by connecting a high voltage power supply form PHYWE HV-Power Supply. Voltage was applied to the spinneret to the surface of water maintained at 10 cm. A flat tip spinneret was used for the flow of polymer solution.

Several parameters were manipulated to study the effect of the parameters on fiber and yarn formation. The voltage applied was varied from 8 kV, 10 kV, 12 kV and 14 kV. The solution flow rate was varied from 0.5 ml/h, 0.75 ml/h, 1.00 ml/h, 1.25 ml/h and 1.50 ml/h. The water velocity was manipulated from 950 L/h, 1000 L/h, 1100 L/h, 1200 L/h and 1450 L/h. The yarn produced which was carried by the falling water from the funnel was collected from the rotating collector inside the water basin.

D. *Nanofiber Characterization*

The morphological structure and fiber diameter of both single fiber and yarn fiber were observed under Scanning Electron Microscope (SEM), Quanta FEG 200. The samples were coated with platinum using a JFC-1600 Auto Fine Coater before viewing under SEM.

III. RESULTS AND DISCUSSION

A. *Morphological Structure of Nanofiber and Nanofiber yarn*

The deposition of fiber in electrospinning process can be on both solid and liquid substrate by following the same principle. In basic electrospinning set-up, the nanofibers were deposited on a large area of the collector surface while in water vortex electrospinning, the fibers are collected from a liquid support system which is water. The parameters affecting the formation and morphological structure of the fiber deposited on solid medium can also be apply to the fiber deposited on liquid medium [14].Fig.2 shows the structure of fiber from water vortex electrospinning and.

The structure of fiber produced from water vortex shows a thread like structure which is called yarn. The formation of yarn was due to the presence of water vortex where the fibers were pulled along with the water down the funnel to the water basin. The spinning force from the vortex resulted in the fibers bundled together forming a continuous yarn. The yarn was collected on the roller collector continuously to produce continuous nanofiber yarn. The velocity of water and roller collector speed had significant effect on the nanofiber alignment in the yarn [14].

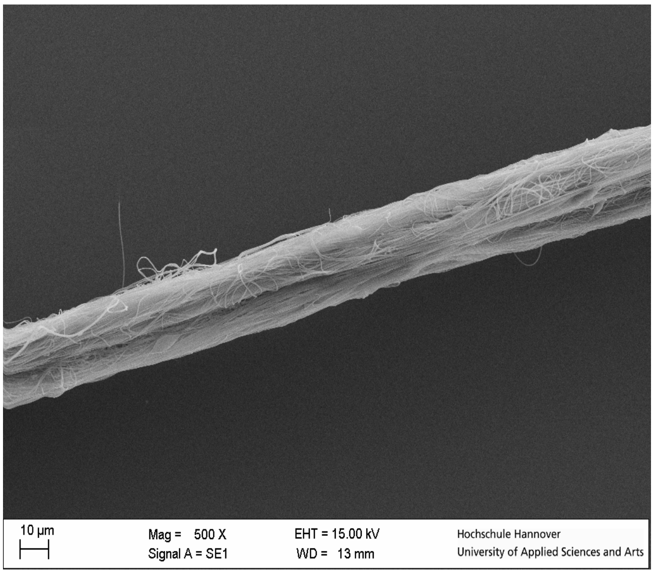

Fig 2: Structure of fiber from water vortex electrospinning

B. *Influence of applied voltage*

The experiment was carried out with the applied voltage varied from 8 to 14 kV. With the increasing of voltage applied, the nanofibers diameter decreases. It can be seen from Fig. 3 the SEM photos of morphological changes of fibers for electrospinning with varied voltage applied. The decreasing fibers diameter were due to increase in electrostatic force resulted from increasing voltage. The electrostatic force on the fluid jet causes the narrowing on of the fiber diameter that made the solution to stretch due to the greater columbic forces in the jet as well as strong electric field. Besides, it also causes rapid evaporation of solvent from the fiber[15]. This was also discussed by Lee et al [16] where increasing the voltage causes the nanofibers become finer.

The effects of applied voltage on the average diameters of fibers were shown in Fig. 3. The single fiber diameter had affect the diameter of the fiber yarn. As the voltage applied increases, the fiber diameter decreases. Therefore, the yarn diameter also decreases. As seen from Fig. 4 shows the fiber yarn diameter from water vortex electrospinning decreased with increasing voltage.

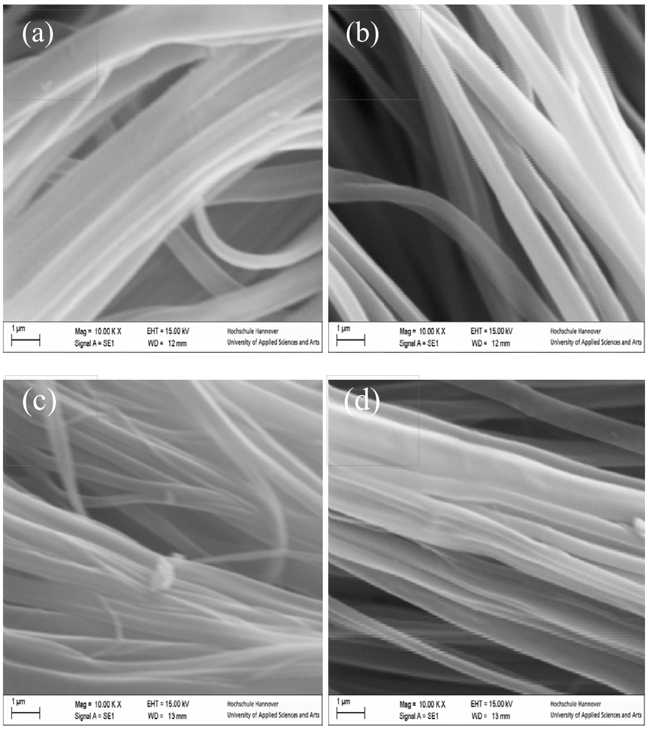

Fig. 3: Water vortex electrospinning (a) 8 kV (b) 10 kV (c) 12 kV (d) 14 kV

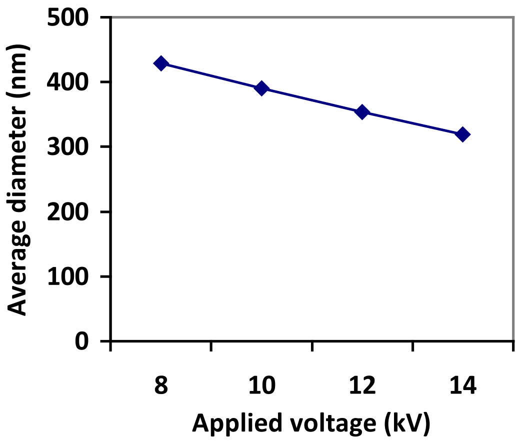

Fig. 4: Graph of average fiber diameter of PAN fiber against voltage applied.

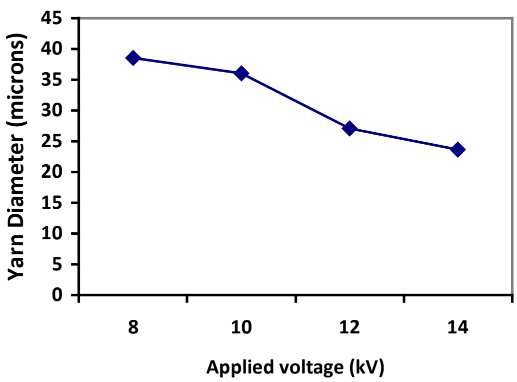

Fig.5: Graph of average yarn diameter of PAN fiber against applied voltage.

C. Influence of solution flow rate

Polymer solution flow rate had a significant effect on the produced nanofiber yarn. The solution flow rate was manipulated at 0.50, 0.75, 1.00, 1.25 and 1.50 ml/h. The other parameters are kept constant at 10kV, 100 rpm roller speed and 1,200 L/h water velocities. When the flow rate solution was increased, liquid droplets are formed at the tip of the needle. This is due to incomplete drying of the nanofiber jet during the flight between the needle tip and the collector[17]. It was observed that the formation of beads occurs when the flow rate is at 1.25 and 1.50 ml/hr. The formation of beads is due to short drying time prior to reach the collector and low stretching forces [18].

Fig 6 shows the formation of beads obtained at 1.25 and 1.50 ml/h solution flow rate. The diameter of the fibers and yarns are also increased linearly with the increasing solution flow rate. Yuan et al., 2004 has also conducted a study where there should always be a minimum flow rate of the solution for the spinning process. It has been observed that the fiber diameter increases with increasing the polymer solution flow rate[19]. The average fiber diameter obtained was 398.48 nm and the yarn diameter was 38.52 µm.

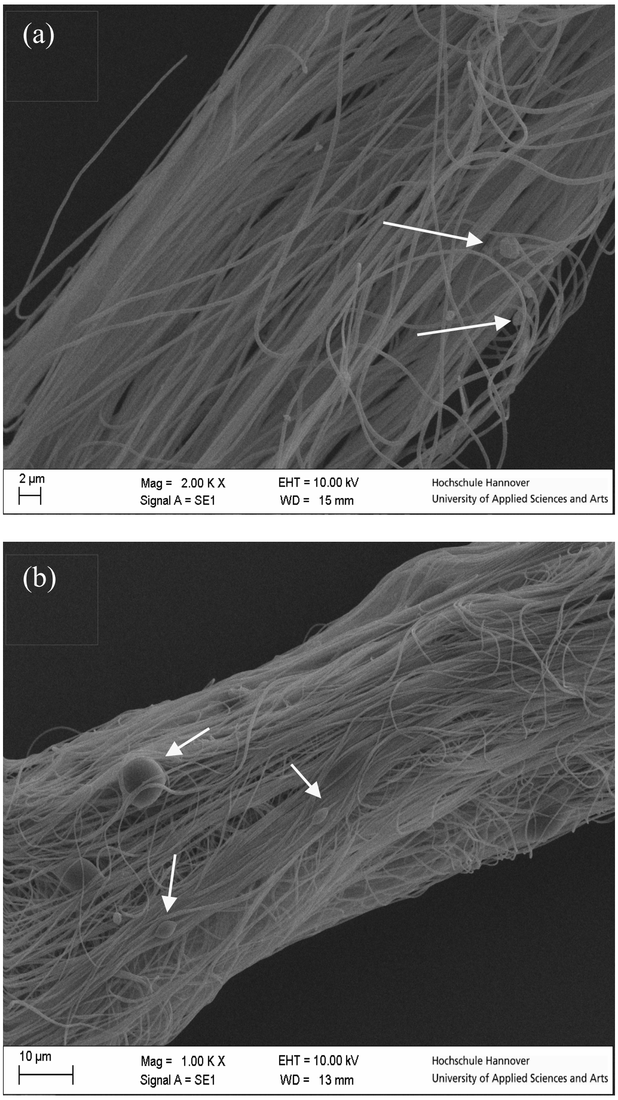

Fig. 6: Formation of beads when the solution flow rate at (a) 1.25 ml/h (b) 1.50 ml/h

D. *Influence of water vortex flow rate*

The speed of flowing water had a significant effect on the fiber formation. The experiment was conducted with manipulating the water velocity at 950, 1000, 1100, 1200 and 1450 L/h. The water speed should be sufficient in order to produce a stable water vortex. Water speed below 950 L/h was unable to produce a stable water vortex and resulted on the fiber entanglement. Observation from the SEM shows a twisting effect on the fiber formation. The twisted angle was the highest at water speed 950 L/h which means the fibers are less twisted.

Some bents of nanofibers in yarn structure were also observed. The occurring bent may due to low water velocity that cause insufficient draft. In addition, breakage of yarn was also observed when the water speed is at the highest speed. The breakage of yarn was due to the high rotational force from the water vortex that the fiber yarn could not withstand the forces[20]. Fig. 7 shows the formation of yarn with twisting effect. Due to breakage of yarn at 1450 L/h water speed, no twisting effect was observed on the fiber yarn. The fiber and yarn diameter and twisting angle decreases with the increasing water velocity. Table 1 shows the measurement of diameter and twist angle of the fiber.

Table 1: Diameter and twist angle relationship with water velocity

Water Velocity (L/h)	Fiber diameter (nm)	Yarn diameter (µm)	Twist angle (°)
950	714.5	94.9	22.4
1000	621.4	81.8	26.1
1200	510.5	47.0	29.0
1450	390.5	36.0	No twist angle

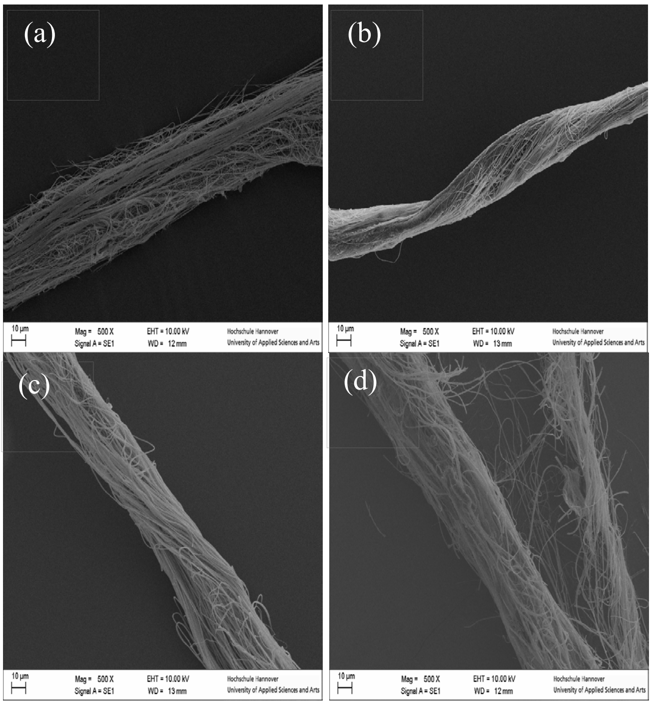

Fig 7: Fiber structure at water flow rate of (a) 950 l/h (b) 1000 l/h (c) 1200 l/h (d) 1450 l/h

IV. Conclusion

Nanofibers were successfully produced using water vortex medium for the electrospinning. The SEM images show the nanofiber structures of water vortex electrospinning in form of nanofiber yarns due to rotating force from water vortex. By manipulating the process parameters, it was observed that the fiber structure and diameter is changing. Further studies can be carried out with the additional of biomaterial and composite material to the polymer to reinforce the formation of fiber. Tensile test can also be conducted to test the strength of the fiber.

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