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Effects of Ambient Temperature and Needle to Collector Distance on PVA Nanofibers Diameter Obtained From Electrospinning Technique

Abstract- Electro spinning is regarded as an active technique for producing biomimetic scaffolds used in tissue engineering applications from synthetic and natural polymers. The technique used in this research gives the ability to produce bio-polymeric materials for fabricating engineered scaffold tissues by preparing (PVA) solution. Ambient temperature at (25, 30, 35, 40, 45 and 50 °C) and needle tip to collector distance with (4, 8, 12, 15, 20 and 22 cm) were studied to optimize the electrospun fibers (size and shape). The electrospun fibers topography were studied by scanning electron microscopy (SEM). Measurements were done for each (SEM) images and lead to determine the mean diameters size of the obtained fibers. Results showed that the average fiber diameter of the (PVA) electrospun decreased to the range (220–500 nm) without creation of any beads, fibers diameter decreased as ambient temperature increase to certain temperature at (45 °C) and retrain to increase at (50 °C) temperature, while increasing the distance of the needle tip to collector decrease the mean nanofiber diameter from (875 nm) at (4 cm) to (600 nm) at (22 cm).

Keywords- Ambient temperature, electrospinning, nanofibers, PVA.

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1. Introduction

Since (1994), the repetition of the term "Electrospinning" in different scientific fields was increased. This term had been driven from the scientific term "electrostatic spinning" [1]. "Cooley" and "Morton" in (1902) were the first whom patented the electrospinning technique [2,3]. In (1934, Formhals) patented his first invention for producing polymer filaments using electric charges [4]. The type of the collecting device of the spinning process is movable type, which collect a stretched thread. This create unwound aligned parallel threads successfully on the collecting device. First spinning patent was by (Formhals's), the patent discuss producing electrospun fibers by using cellulose acetate polymer and acetone solvent [5]. This first spinning method has some technical disadvantages because of the short distance between the nozzle tip and the collection plate. Drying fibers completely was very difficult due to short distances. (Formhals) subsequent patent, solve the obstacles by changing the nozzle and collection zones distance which increase the time for drying the electrospun fibers and the formation process of them. Subsequently in (1940), another patent discuss a method for producing composite web fibers from different kinds of polymers by electrostatically spinning procedure to polymer fibers on a moving base substrate [6]. In (1966),

(Simons) patented an apparatus produce ultra-thin and very light in weight non-woven fabrics by electrospinning technique. Producing (finer and shorter) electrospun fibers can be done by using low viscous solutions while more viscous solutions produce continuous electrospun fibers [7]. In (1969), (Taylor) investigated the droplet shape of the polymer produced at needle tip subjected to an electrical field. (Taylor) refer to a creation of a cone from the polymer droplet and jets will be ejected from cone vertices. Different kinds of viscous fluids were investigated by (Taylor) and results showed that at an angle of (49°), a balance for the surface tension of the polymer with the electrostatic forces is very required. In addition, (Taylor) referred to the importance of shape's jet, which must be conical because of its major effect on the beginning of the gradients extensional velocity which effect on fiber forming processes [8]. Conical jet shape was later known as the "Taylor cone".

Electrospinning is the cheapest and the most straightforward way to produce nanomaterials. For several kinds of applications, structured polymer fibers which their diameters varies from micrometers to tens of nanometers are considered to be important in the production operations of these applications. The possibilities of producing fibers from a biodegradable and renewable product

had become easier with low cost and high strength fiber properties to be used in different fields in life. The electrospinning operations affiliate the electrostatic spraying operation and uses it. Such method approaches the processes of using polymer biomaterials with the opportunity of controlling its morphology, porosity and composition by using simple device. Materials in nanofiber form have an exceptionally high specific surface area, this gives the ability to high proportion of atoms to create the fiber surface and be on it. This will improve the quantum efficiency, the surface reactivity, the thermal conductivity and the strength of the fibers produced [9].

One of the unique characteristic of electrospinning process that it can provide an easy and controlled method to produce nanofibers. Nanofibers are desired because they have a large surface area to volume ratio. High surface area provides more area to bind virus and can achieve higher virus removal and larger membrane capacities. This ratio can be as high as (103) times to typical commercial microfilters. Nanofibers can also provide flexibility in their surface functionalities and high tensile strength. All these benefits made nanofibers an excellent choice for biological application [10]. The polymeric fibers produced by electrospinning process can produce fibers with micrometer diameter range of (10-100 μm) and can reduce the diameter to sub-micron or nanometer with the range (0.01-0.1 μm) which create some desired characteristics like: high surface area to volume ratio (reaches (103) times of microfibers ratio), high porosity and high pore size in (nano range) [11]. The aim of the research was to investigate the influence of selected parameters like the ambient temperature and the distance between the fibers collector and the tip of the fiber-ejected needle on fiber formability.

2. Materials and Methods

I. Characteristics of Polyvinyl alcohol (PVA)

Polyvinyl alcohol (PVA) used greatly in world's industries in different fields. The solubility of the polymer in water and many of its advance and good synthetic properties had made the polymer became a part of several kinds of engineering product, like the nonhazardous property which made it used in many adhesives. Preparation of Polyvinyl Alcohol (which its density is (1.31 g/cm^3), its molecular weight (80,000)) was by (Sinopharm chemical Reagent Co.). In this research, the solution was prepared by mixing ((10 gm) of (PVA) powder with (90 ml) of distilled water) as shown in Figure 1.

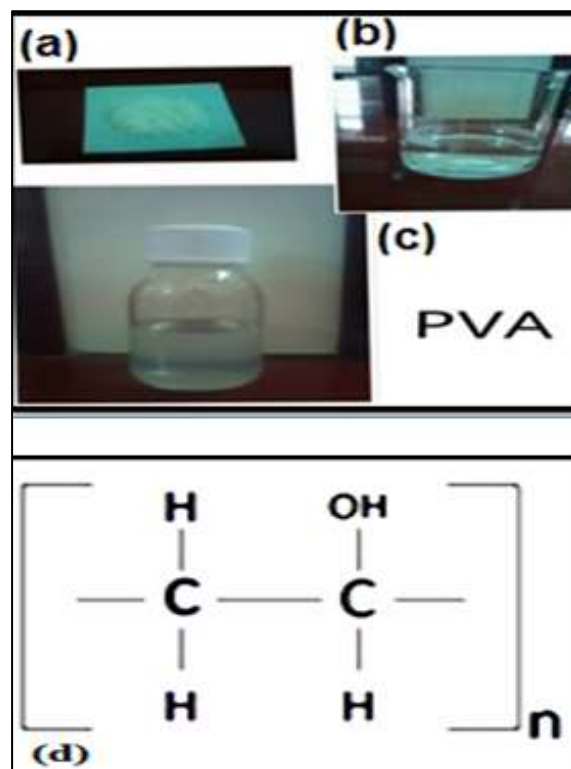


Figure 1: (a, b and c) represents the PVA preparation: (a) powder, (b) distilled water and (c) PVA solution respectively, (d) Chemical structure of (PVA) which is represented by $(\text{C}_2\text{H}_4\text{O})_n$ formula

Important tests for prepared (PVA) polymer solution before spinning process carried by some devices, these tests like: Electrical conductivity measured by using (Cand 7110 inolab) device, viscosity measured by using (Viscometer (DV-II+Pro) Brook field) device and measuring surface tensiometer Model (using JYW-200A LARYEE TECHNOLOGY CO.).

II. Electrospinning process

The prepared electrospun solution was gathered in (10 mL) syringe equipped with a (22-gauge) stainless steel needle tip. The electrospinning process was carried out by a bio-electrospinning/electrospray system (ESB-200), provided by (Nano NC, South Korea) at Fibers Technology Lab-Dept. of Materials Eng. University of technology, which is shown in Figure 2. After fixing the plastic syringe (10 ml) on to the pump, the pump was turned on, to provide (0.2 ml/hr), the polymer solution started flowing out of the needle (with orifice size 0.6 mm). A voltage was applied on the solution; the voltage was increased until a jet of polymer solution ejected from the tip of needle. The voltage was further adjusted to stabilize the (Taylor cone) and the jet at (15 KV). Studying the ambient temperature with (25, 30, 35, 40, 45 and 50 $^{\circ}\text{C}$) and needle tip to collector distance with

(4, 8, 12, 15, 20 and 22 cm.) were done then to determine the influence of these technological parameters on fibers formability. After finished experiments, the samples prepared were remove from collector and kept at laboratory ambient temperature for (24 hours) to achieve the elimination of solvents.

The stand of the device is composed of three basic elements: a high voltage generator, an upper electrode and a lower electrode. The upper electrode serves for extruding the polymer, which enables the polymer drops to reach the suitable electric potential. The second (lower) electrode is the take-up electrode, which the electric potential of the polymer is applied and the fibers are deposited during the process of manufacturing the nonwoven. Figure 2 represents a photograph of its main parts and a photograph of the electrically driven bending instability of the jet.

III. Analysis of electrospun fibers

Electrospun fibers morphology analysis were done. Samples of the electrospun fibers were sputter-coated with (Au) and examined with a scanning electron microscope (SEM, VEGA). All micrograph from the (SEM) scans were analyzed statically. Average diameter of the electrospun fibers were calculated and drawn as histogram figures to obtain the fibers diameter distribution and their average.

3. Experimental Results

I. Characteristics of Polyvinyl alcohol (PVA)

Testing the (PVA) water solution properties like: Electrical conductivity was (1222 $\mu\text{S}/\text{cm}$), viscosity was (2014 Cp) at (19 °C) and surface tension was (44 mN/m²).



Figure 2: (a) Nano bond electrospinning system with rotation collector, (b) with flat plate collector, (c) needle connected to high voltage by upper electrode and (d) flat aluminum plate collector connected to grounded lower electrode with Taylor cone and spun image

II. Electrospun fibers characterization

The factors effecting on the shape and size of the electrospun fibers are: polymer solution, applied voltage air gap distance between the needle tip to collector, ambient temperature and the used flow rate. These factors controls the electrospun fibers shape and size. Ambient temperature and needle tip to collector distance were studied to reach electrospun fibers with a desirable production. To investigate ambient temperature effect, the solution flow rate was kept constant at (0.2 ml/hr), the voltage applied on the solution was kept at (15 KV). Figure 3, 4 and 5 illustrates the (SEM) images of nanofibers obtained at different ambient temperature of (25, 30, 35, 40, 45 and 50 °C) with histogram of each them respectively. The average fiber diameter was found to decrease from (425 nm) at a temperature of (25 °C) to (220 nm) at a temperature of (45 °C) then return to increase to (416 nm) at (50 °C), it was noticed that with increasing temperature high uniformity and homogeneity of nanofibers will be produced and make nanofibers to take nearly narrow range of diameter due to high evaporation of solvent. It was found that fiber diameters are temperature dependent and by increasing temperature, average fiber diameter decreased. But when decreasing the ambient temperature will reduce the evaporation rate of solvent and give longer solidification time of the jet and all lead to decrease the average fiber diameter, fiber formation will be optimum when the solution is delivered to the collector at the ambient temperature of (45 °C) [12,13]. Figure 6 shows relationship between ambient temperatures and fibers diameter that increased in temperature decreased fiber diameter until (45 °C) which can be considered as a temperature critical value then return to increase at (50 °C), may be due to fibers adhesion between them after collection in collector.

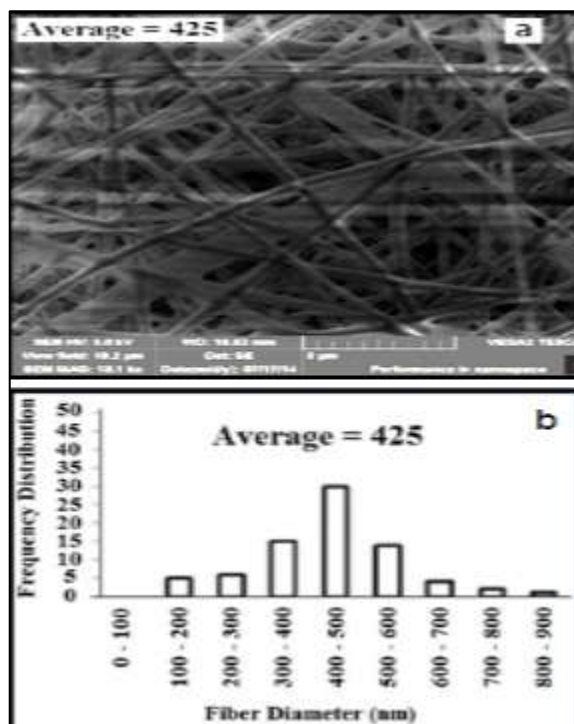


Figure 3: (a) SEM images of nanofibers obtained at ambient temperature 25 °C and; (b) its Histogram

The needle tip-collector distance effects on the electrospun fibers size and the periodicity of biopolymer fibers were also studied. Some parameters like: concentration of (PVA), applied

voltage were kept constants at (15 kV) respectively.

According to (SEM) images of nanofibers obtained at different distances as shown in Figures 7, 8 and 9 with histogram of each them, it could be noticed that by altering the distance between tip to collector, the average fiber diameter will decrease from (875 nm) at distances of (4 cm) to (250 nm) at distance of (20 cm) as decreasing diameter by increasing distance up to (20 cm), increasing the tip-collector distance over (20 cm) will increase the diameter of the fibers. Possibly the larger distances increase ejection time tip-collector of the jet, stretching of the solution so the solvent had enough time to evaporate completely. Dried fibers stretched and deposited on the collector, lead to result in reducing the diameter. It was noticed that, by placing collector at distance of (8 cm) and lower from needle tip, some fibers were seen but most of them got coalition [14-16]. The reason would probably be due to the decrease in ejection time, tip-collector and lack of enough time for excess solvent evaporation when the jet reached the collector. By placing the collector at a distance of (22 cm), some of spun fibers may fail to collect on the collector. That conclude as too small distance can lead to produce “wet” fibers that fuse on the collector.

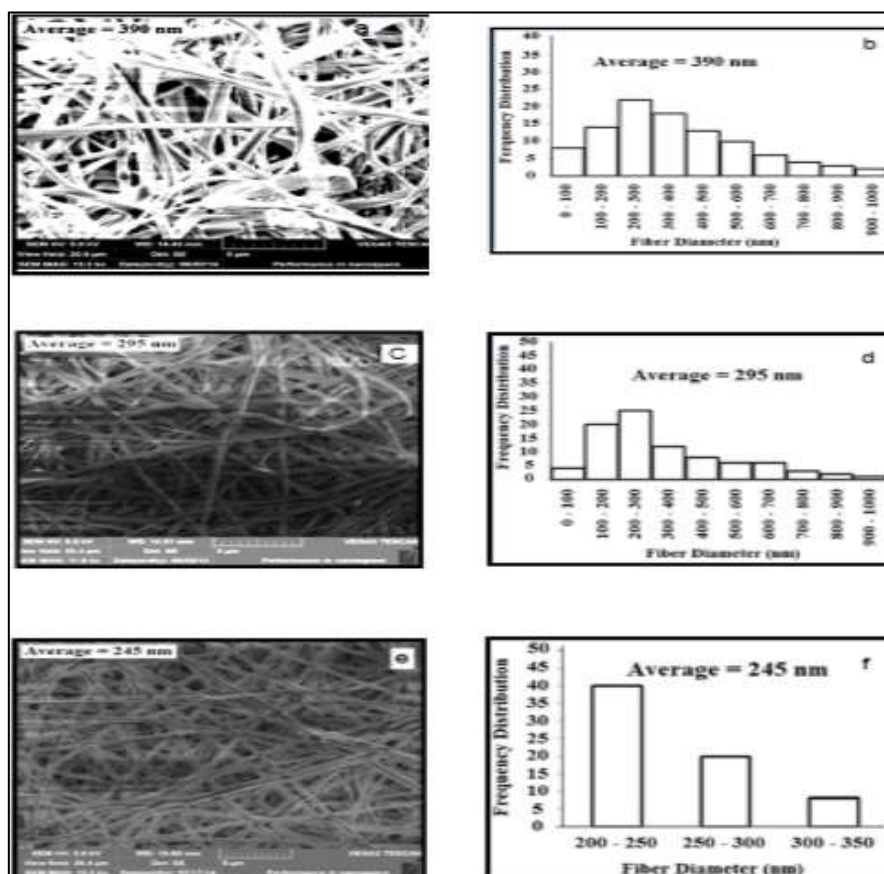


Figure 4: SEM images of nanofibers obtained at different ambient temperature of (a) 30, (c) 35, (e) 40 °C with (b, e, f) Histogram of each them respectively

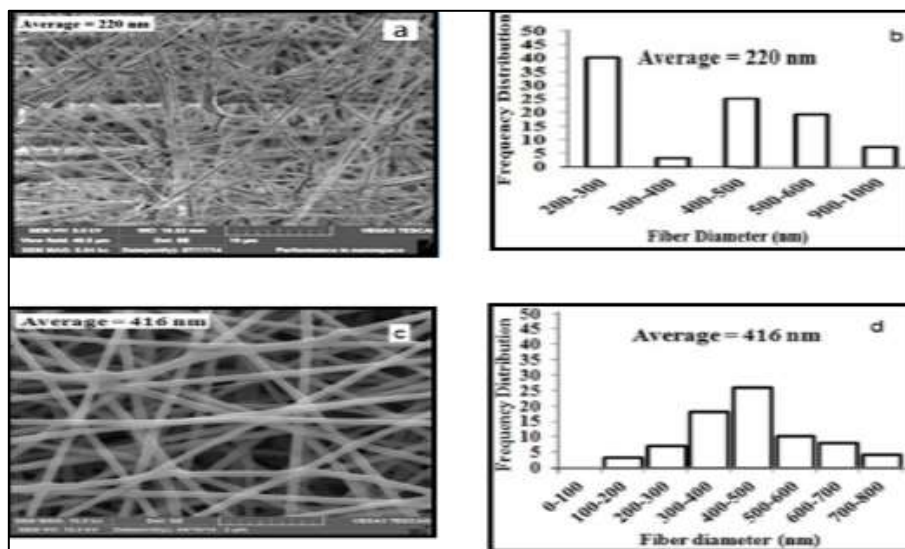


Figure 5: SEM images of nanofibers obtained at different ambient temperature of: (a) 45 and (c) 50 °C (b, d) with Histogram of each them respectively

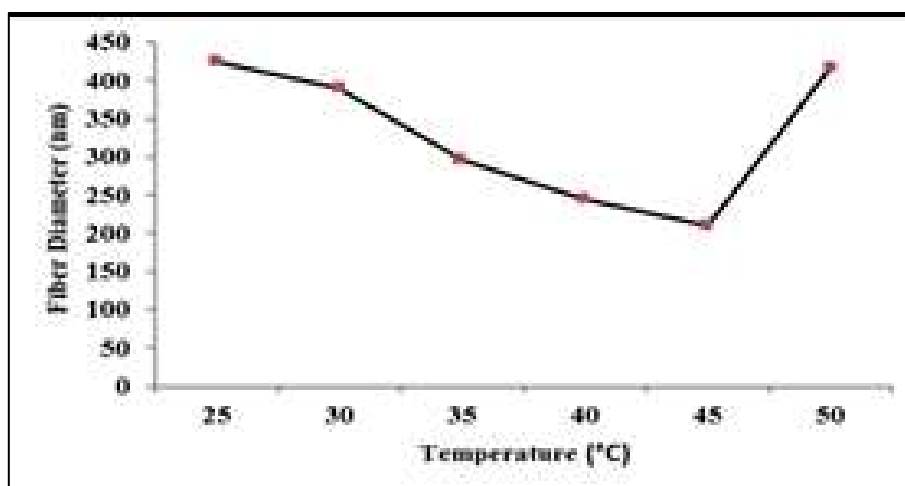
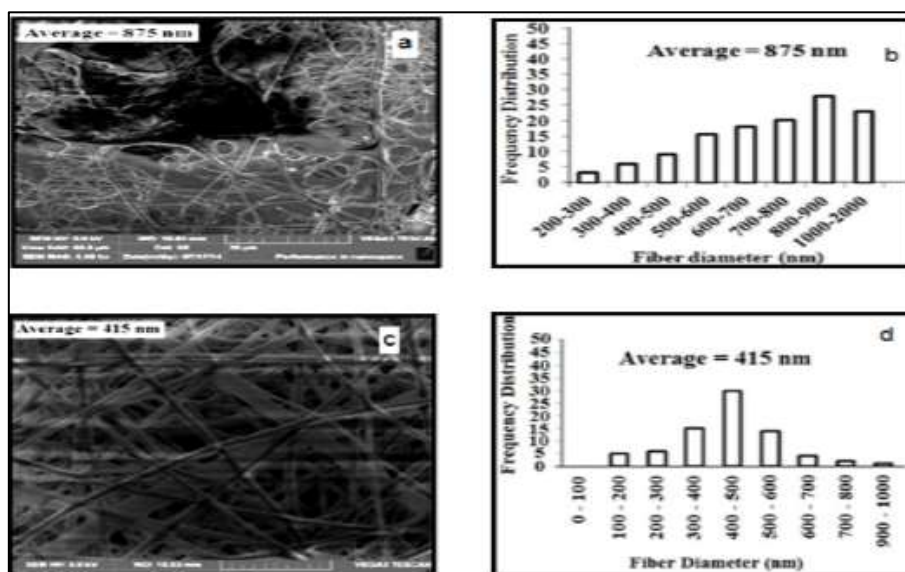
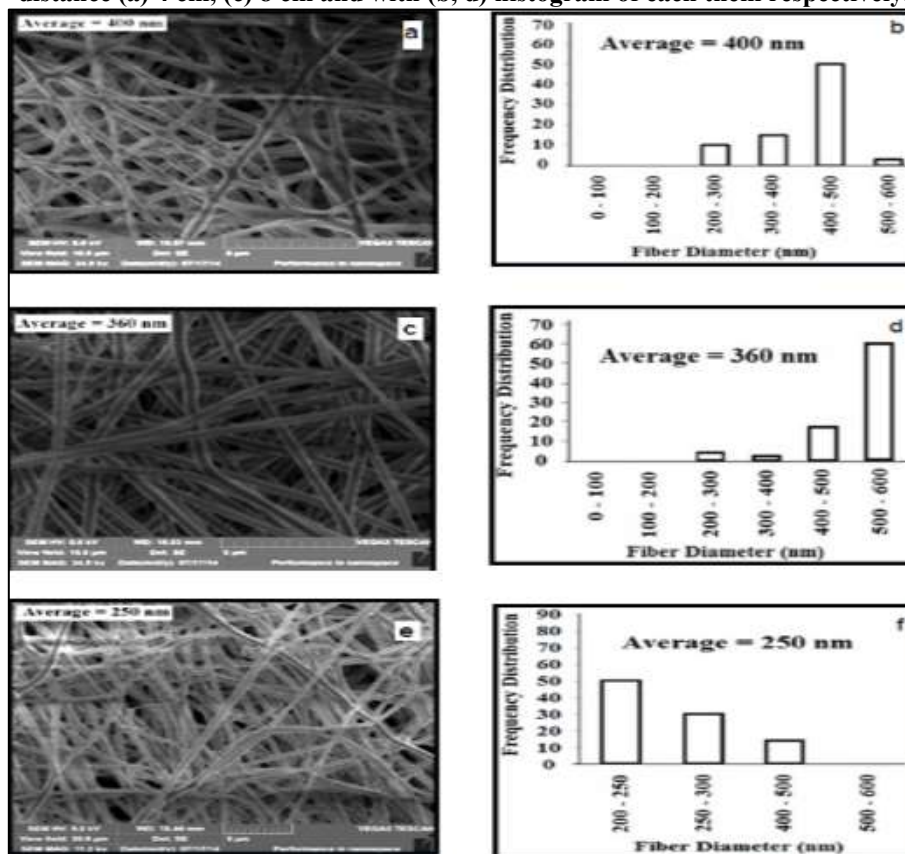


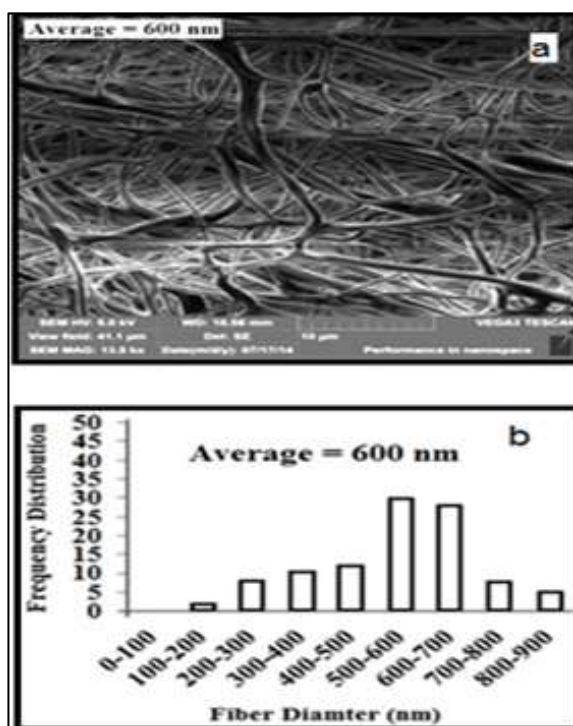
Figure 6: Distribution of Nanofiber with various Temperatures



Figures 7: SEM images of (PVA) nanofibers at (15 KV), flow rate (0.2 ml/hr) at different needle tip-collector distance (a) 4 cm, (c) 8 cm and with (b, d) histogram of each them respectively.



Figures 8: SEM images of (PVA) nanofibers at (15 KV), flow rate (0.2 ml/hr) at different needle tip-collector distance: (a) 12 cm, (c) 15 cm, (e) 20 cm with (b, d, f) histograms of each them respectively



Figures 9: SEM images of (PVA) nanofibers at (15 KV), flow rate (0.2 ml/hr) at needle tip-collector distance (a) 22 cm with (b) histogram

When the time for the solvent to evaporate increased, dry solid fibers are collected at the target. With increasing the distance between the needle-tip and the target, the jet underwent a larger amount of electrically driven bending or whipping instability. It was found that the optimum collection distance is (20 cm). Consequently, the amount of stretching or elongation of the jet increased which leads to decrease the fiber diameter and that match with references [17-21].

As shown in Figure 10 increasing in distance of needle tip-collector decreases fiber diameter but to certain distance which be a critical distance (20 cm), after it fiber diameter return to increase.

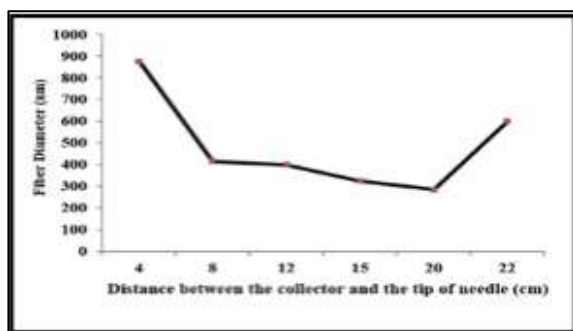


Figure 10: Distribution of Nanofiber with various Distances

4. Conclusion

As the air gap of needle tip to collector distance increased, solvent evaporation time increased and this lead to collect dry solid electrospun fibers on the collector successfully. Consequently, stretching or elongation of the jet will increase and that leads to decrease the electrospun fiber diameter, also the pore size decrease with increase collection distance.

By increasing ambient temperature, average electrospun fiber diameter decreased. Nevertheless, that decrease will create a reduction in evaporation rate of the solvent and solidification time of the jet will increase and all lead to a decrease in average electrospun fiber diameter.

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