Study the Effect of Electrospinning Device Room Temperature on Electro-spun PVA Nano-fibers

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Abstract

This research focuses on studying the effect of electrospinning device room temperature on PVA nano-fibers diameter made by electrospinning device depositing on rotating drum. In this research, the preparation of nano-non woven fabrics by electro-spun polymer solution of poly vinyl alcohol using distilled water as solvent in temperature range [15-35]C° was achieved. The relative humidity 40%, polymer solution concentration 14% wt., voltage 22KV, tip-to-collector distance 10 Cm. The results of the five scanned samples by scanning electron microscope (SEM) indicated that: by increasing room temperature the diameter of nano-fibers decreased. Also it is important to mention that the range of room temperature shouldn't be exceeded in order not to close the orifice of the needle because of the evaporation of the droplet solvent changing into solid polymer.

Keywords: Electrospinning, Nano-fibers, Temperature, Poly vinyl alcohol (PVA)

Introduction:

Nano fiber is defined as the fiber having at least one dimension in nanometer range [1]. Nano-fiber is used for a wide range of medical applications for drug delivery systems, scaffold formation, wound healing and widely used in tissue engineering, skeletal tissue , bone tissue, cartilage tissue, ligament tissue, blood vessel tissue, neural tissue etc [2]. It is also used in dental and orthopedic implants. Electrospinning is a process that produces a highly impermeable, nonwoven fabric of submicron fibers by pushing a millimeter diameter liquid jet through a nozzle with an electric field, it is a process in which solid fibers are produced from a polymeric fluid stream solution or melt delivered through a millimeter-scale nozzle. The solid fibers are notable for their very small diameters <1 µm [3]. General electrospinning setup shows in figure (1).

Numerous parameters act on the electrospinning process which affects the resulting nano-fiber characteristics. Due to the difficulty of precisely controlling the large number parameters the reproducible production of nano-fibers becomes problematical which is an issue for the use and development of nano-fibers in industries such as biotechnology & health care. These parameters can be split up into three subcategories:

Polymer solution parameters involving rheological and chemical properties of solutions.

Processing conditions which include applied voltage, flow rate, spinneret and collector properties. Ambient parameters where atmospheric conditions interact with the system to affect fiber morphology. Varying any of these parameters even by small amounts can have a large effect on the structure of fibers produced; this enables the formation of fibers with defined features such as fiber diameter, flat ribbon or cylindrical fibers, level of fiber surface porosity and bead formation. For instance, the humidity of the electrospinning environment plays a part in the formation of porous nano-fibers. However, there are other factors that may contribute to the formation of pores on nano-fibers during electrospinning. Also the solvent vapor pressure has a critical influence on the process of pore formation. The mechanism by which the flattened or ribbon-like fibers were obtained can be related to solvent evaporation during electrospinning process. Branched fibers are produced due to the elongation of the jet and evaporation of the solvent alter the shape and the charge per unit area carried by the jet. The balance between the electrical forces and surface tension can shift, thereby causing the shape of a jet to be unstable. Such instability can decrease its local charge per unit surface area by ejecting a smaller jet from the surface of the primary jet or by splitting apart into two smaller jets.

Depending on the intended application these properties have the potential to be selected and specifically expressed. For example thinner fibers may be preferred due to the larger surface area that they convey but with small diameter comes an inherent reduction in fiber strength [4]. Aligned and random fiber mats can be formed by changing the collector plate and by manipulating the external electric field [5].

Nano-fibers prepared by electrospinning have several advantages, such as large surface area to volume ratio, high specific surface area and small pore size, superior mechanical properties and flexibility in surface functionalities [6]. In the present work a water-soluble synthetic polymer Poly (vinyl alcohol) (PVA) was used. The aim of this research is to evaluate the influence of temperature effect on the diameter of nano-fibers produced by our electrospinning device.

Some previous researches also discussed this effect, when polyurethane is electro-spun at a higher temperature, the fibers produced have a more uniform diameter. This may be due to the lower viscosity of the solution and greater solubility of the polymer in the solvent which allows more even stretching of the solution [7]. For Polyarnide-6 with a low viscosity, the Columbic forces are able to exert a greater stretching force on the solution thus resulting in fibers of smaller diameter. Increased polymer molecules mobility due to increased temperature also allows the Columbic force to stretch the solution further [8]. In the preliminary studies of the effect of temperature on PVA nano-fibers morphology, the morphology of the fibers were found to be "beaded" for the low temperature(<40C°), but at higher temperatures, the fibers became flat. According to their determinant parameters [9]. In another research, continuous nano-fibers were prepared by bubble-electrospinning of poly(vinyl alcohol) (PVA) and poly(vinyl pyrrolidone) (PVP) solutions at different temperatures between 25 °C and 75 °C, respectively. The results show that the average diameter of the fibers decreased with the temperature increase [10].

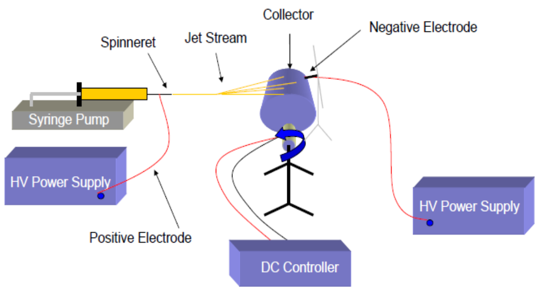


Figure 1: General electrospinning set-up [11]

Experimental

1. Materials:

a- Poly vinyl alcohol (PVA), Available in a finely divided form, its degree of polymerization (1800-1700), degree of hydrolysis (98-99%), is made in MUMBAI-INDIA.

b- Distilled water, made by our laboratory distillation unit.

2. Procedure:

PVA solutions were prepared by dissolving PVA into distilled water at 85°C, where 14g PVA added into 86 ml distilled water, stirred well for an hour.

Electrospinning apparatus (fig.(2)) used in this study consists of high voltage power supply, pump, syringe, syringe needle, electrospinning device room temperature regulator, lamps, fans, electrospinning device room humidity regulator and rotating collector.

The solution prepared was put into a syringe for electrospinning at room temperature while the syringe needle was inside electrospinning room. The collector is grounded, syringe needle is connected with high voltage power supply electrode and fixed with the syringe which is in its turn fixed with the pump adjusted flow rate 4ml/h.

A small drop of polymer solution comes out of syringe needle which can be pulled by strong electric field toward the collector and solidified on it to form nano-fibrous structure (nano-fibers).

Five experiments have been done with five different electrospinning room temperature but same else parameters as: distance between syringe needle and collector (d=10 Cm), voltage supply (V=22kV), relative humidity (R=40%), polymer concentration (C=14%wt.), polymer flow rate (F=4 ml/h).
The five electrospinning room temperatures are:15,20,25,30,35 °C.

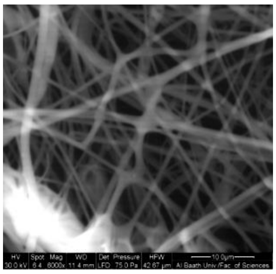
Five samples (non woven nano-fibers mats) were prepared then scanned under scanning electron microscope (SEM) and analyzed using Imagej program to get the average nano-fibers diameter for each sample at each different electrospinning room temperature.

Figure 2: Electrospinning apparatus used at this research

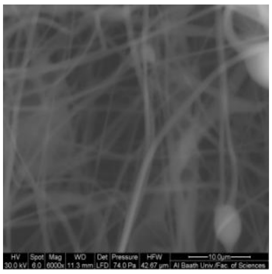
Results and Discussion

The results are taken from the electro-spun non woven nano-fibers mats fabricated under specified parameters mentioned above.

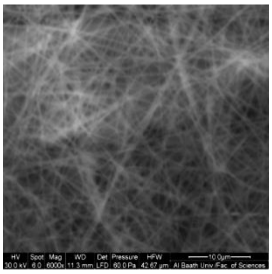
Non woven nano-fibers mats produced by electrospinning device shown in figure (3) using amplification 6000x.



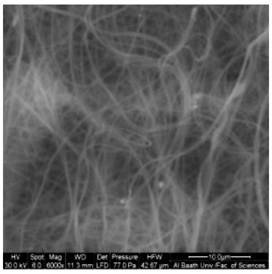
At electrospinning room temperature15 °C



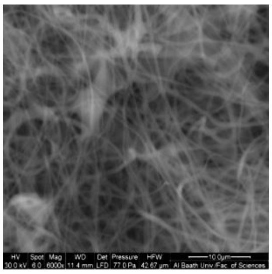
At electrospinning room temperature 20 °C



At electrospinning room temperature 25 °C



At electrospinning room temperature 30 °C



At electrospinning room temperature 35 °C

Figure 3: SEM photos of non woven nano-fibers mats samples prepared at different electrospinning room temperatures

The average diameter of each sample was measured and calculated, as listed in table (1).

Table 1: the values of average nano-fibers diameter at different electrospinning room temperatures

Average nano-fiber diameter (Nm)	Electrospinning room temperature (°C)
562.3	15
509.8	20
382.9	25
264.6	30
253.5	35

From the above table, while the temperature of electrospinning device room increases the average diameter of nano-fibers decreases.

The explanation of the reason of the effect can be due to the fact that increasing temperature, reducing the viscosity of the polymer solution. The lower viscosity of the polymer solution allows more even stretching of it [7]. With a lower viscosity, the columbic forces are able to exert a greater stretching force on the solution thus resulting in fibers of smaller diameter. Increased polymer molecules mobility due to increased temperature also allows the Columbic force to stretch the solution [8]. Generally, when the temperature of the solution is increased, the solubility of the polymer in the solvent will increase. In pure solvents, specific viscosity decreases with increase in temperature. This is due to higher polymer chain mobility. The dependency of intrinsic viscosity on temperature can be shown by[4],

$$[\eta] = Be^{E_a/RT} \quad \dots (1)$$

where η is intrinsic viscosity
E_a is an activation energy for viscous flow
B is a constant
R is the ideal gas constant
T is the temperature in Kelvin.

Also it is important to mention that working at electrospinning device room temperature 35°C causes needle clogging after few minutes of working so this temperature is not desired and working at electrospinning device room temperature more than 35°C causes a direct complete evaporation for the solvent of the droplet leaving a small piece of solid polymer which will close the tip of the needle and electrospinning process couldn't be done.

It was noted from Figure (3) that the most uniform nano-fibers having the least amount of beads were at the temperature 25°C.

It is important to mention that the increased temperature the reduced beads which happened because of reduced viscosity of the polymer solution (due to increased temperature) allowing for a more even stretching due to the more dominant effects of the columbic forces.

Many experiments have shown that a minimum viscosity for each polymer solution is required to yield fibers without beads [12],[13]. And one of the methods to minimize viscosity is to increase the temperature.
Both of reducing beads and decreasing in average nano-fibers diameter are usually desirable for a lot of applications.

Conclusion

PVA nano-fibers were electro-spun under a controlled temperature. The effects of electrospinning device room temperature on nano-fibers were investigated. The result obtained by SEM indicated the impact of: with increasing process temperature, the average of nano-fibers diameters and beading effect decreasing. The average fiber diameter decreased from 562.3 Nm to 253.5 Nm for process temperatures 15°C and 35°C respectively. Electrospinning room temperature for nano-fiber production in this work which yield the most suitable fiber mats for a membrane purpose are 25°C because of the highest level of fiber diameter uniformity.

References

[1] Chuangchote, S., Supaphol P., (2006), "Fabrication of Aligned Polyvinyl Alcohol Nano-fibers by Electrospinning", Journal of nano-science vol. 6, 125-129.

[2] Rebicek, J., Pokorny, M., Velebny, V., (2011), " Aligned nano fiber deposition onto a patterned rotating drum collector by electrospinning", Brno, Czech Republic, EU, 21-23.

[3] Hohman, M., Shin, M., Rutledge, G., Brenner, M., (2001), " Electrospinning and electrically forced jets. I. Stability theory", American Institute of Physics, Phys. Fluids, vol. 13, 2201-2220.

[4] Seeram Ramakrishna, Kazutoshi Fujihara, Wee-Eong Teo.Teik-Cheng Lim & Zuwei Ma (2005), " An Introduction to Electrospinning and Nano-fibers ". World Scientific Publishing Co. Pte. Ltd., National University of Singapore p.(118-127), (72).

[5] Park, S., Park, K., Yoon, H., Son, J., Min, T., Kim, G., (2007), " Apparatus for preparing electro-spun nano-fibers: designing an electrospinning process for nano-fiber fabrication", Polymer International Journal, vol. 56, 1361-1366.

[6] Huang, Z., Zhang, Y., Kotaki, M., Ramakrishna, S., (2003), " A review on polymer nano-fibers by electrospinning and their applications in nano-composites", Composites Science and technology, vol. 63, 2223-2253.

[7] Demir, M. M., Yilgor, I., Yilgor, E. and Erman, B. (2002), "Electrospinning of Polyurethane Fibers". Polymer. Vol. 43, p. 3303-3309.

[8] Mit-uppatham, C, Nithitanakul, M. and Supaphol, P. (2004). "Ultrafme Electrospun Polyarnide-6 Fibers: Effect of Solution Conditions on Morphology and Average Fiber Diameter". Macromol. Chem. Physic. Vol.205, p. 2327-2338.

[9] Didem R., Mehmet M.(2012), " Effects of Electrospinning Setup and Process Parameters on Nanofiber Morphology Intended for the Modification of Quartz Crystal Microbalance Surfaces". Journal of Engineered Fibers and Fabrics. Vol.7, p.118-123.

[10] Qiu-Na ., Ya L., and Fu-Juan L. (2014), " Effect of The Temperature on The Morphology of Bubble-electrospun PVA And PVP Nanofibers". Thermal Science.Vol.15,No.5, p.1707-1709.

[11] Carnell, L., Wincheski, R., Siochi, E., Holloway, N., (2007), " Electric Field Effects on Fiber Alignment using an Auxiliary Electrode during Electrospinning ", National Aeronautics and Space Administration, MRS Conference.

[12] Megelski, S., Stephens, J.S., Chase, D.B. and Rabolt, J.F. (2002). "Micro- and nanostructured surface morphology on electrospun polymer fibers", Macromolecules. Vol.35, p. 8456-8466.

[13] Fong, H., Chun, I., Reneker, D. H. (1999). "Beaded nanofibers formed during electrospinning". Polymer.Vol. 40, p. 4585-4592.

دراسة تأثير درجة حرارة حجرة جهاز الغزل الكهربائي على ألياف بولي ڤينيل الكحول المغزولة كهربائياً

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الخلاصة:

يركز هذا البحث على دراسة تأثير درجة حرارة حجرة جهاز الغزل الكهربائي على قطر ألياف بولي ڤينيل الكحول النانوية المصنعة بوساطة جهاز الغزل الكهربائي والمترسبة على مجمع دوار. في هذا البحث، تم الحصول على أقمشة غير منسوجة مكونة من ألياف نانوية بوساطة محلول بولي ڤينيل الكحول الخاضع لعملية الغزل الكهربائي باستخدام الماء المقطر كمذيب وذلك ضمن مجال من درجات الحرارة يتراوح من 15 م° وحتى 35 م°. الرطوبة النسبية 40%، التركيز الوزني للمحلول البوليميري 14%، الجهد الكهربائي 22 كيلو فولط، المسافة بين المجمع ورأس إبرة الغزل 10 سم. إن نتائج العينات الخمسة المفحوصة باستخدام المجهر الإلكتروني أشارت إلى أن: زيادة درجة حرارة الغرفة تؤدي إلى تناقص قطر الألياف النانوية. كما تجدر الإشارة إلى أنه لا يجب تجاوز مجال درجة الحرارة كي لا يتم إغلاق فوهة الإبرة بسبب تبخر المذيب الموجود في القطرة وتحولها إلى كتلة بوليميرية صلبة.