

Fabrication of Chitosan Nanofibers Membrane *via* Electrospinning

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ABSTRACT

Chitosan nanofibers were prepared *via* electrospinning by varying solution concentration (1, 2, 3, 4 and 6 wt %), applied voltage (14, 18 and 22 kV), flow rate (0.4 mL/h and 0.8 mL/h), needle diameter (0.2 mm and 0.8 mm) and distance between needle tip and collector (100 mm and 140 mm). No electrospinning was observed for 1-3 wt% samples, where as improved physical texture, decrease in the number and size of the beads and microspheres (in case of 4 wt%), and beads free nanofibers were obtained when the concentration of solution was changed from 4 to 6 wt%, applied voltage, flow rate and needle diameter from 14 kV, 0.4 mL/h, 0.2 mm to 22 kV, 0.8 mL/h and 0.8 mm, respectively. Increase in the polymer concentration has increased the viscosity of the solution to a critical value, higher voltage overcame the surface tension of the polymer solution, changes in the flow rate increased the flow rate above the threshold and change in needle made the diameter large enough to avoid evaporation of solvent at the needle tip, its blocking and allow the formation of jet. The effect of solution flow rate, distance between the needle tip and collector, and needle diameter on the morphology of nanofibers was observed only in case of 4 wt%. In all three cases the number and size of the beads decreased.

Keywords: Chitosan, Electrospinning, Optimization, Nanofibers

1. INTRODUCTION

Electrospinning is a unique technique for producing fibers (using electrostatic forces) with diameters between 10 and 200 nm nanometers. This technology traces its roots back to 1902 and 1934 when the first patents were registered on the electrical dispersion of fluids and fabrication of textile yarns from electrically dispersed fluids [1]. However, this technique did not receive much attention until the 1990s, when an expansive range of organic polymers were electrospun with nano-sized diameters, since

then interest in this science was revitalized and the number of publications on electrospinning has increased dramatically each year [2-4]. Today, electrospinning is an established technique for generating nanofibers [3,5-7]. A typical electrospinning setup usually includes a reservoir of polymer solution with a metallic capillary connected to high voltage and a metallic collector. The hemispherical shape of the droplet at the needle tip is destabilized by the accumulated charges on the surface, and is converted to a Taylor's cone, when high voltage is applied to a polymer solution. At a critical value of the voltage, the electric forces overcome the surface tension on the droplet and a jet of ultra-fine fibers is produced from the tip of the Taylor cone [4]. Nanofibers, produced *via* electrospinning provides a facile and an effective mean in producing nonwoven fibrous materials [8] with interesting characteristics such as fine diameter [9], large surface area per unit mass, high porosity, high gas permeability, and small interfibrous pore size [10]. Any soluble polymer with sufficient molecular weight to achieve a critical viscosity can be electrospun; however, the preparation of fine nanofibers requires careful consideration of many operating parameters (such as polymer molecular weight, applied voltage, solution flow rate and spinning distance), environmental parameters (such as temperature, humidity and air velocity in the chamber) and solution properties (such as conductivity, viscosity and surface tension). Polysaccharides nanofibers has been intensively studied due to their abundance in nature, cheap availability, and potent industrial applications such as novel separating [7] and scaffolding materials for tissue cultures [11]. However, at present very limited reports on the electrospinning conditions of the polysaccharides are available in the literature. In the present work we will undertake the effect of solution concentration, applied voltage, solution flow rate, needle diameter and spinning distance. We believe that the present study will not only prove a potential step forward in improving the morphology and properties of previously available nanofibers but will so find more novel applications such as gas sensors, nano-electronics and nanocatalysis, etc.

2. EXPERIMENTAL

2.1. Materials and Methods

Medium molecular weight chitosan powder and Trifluoroacetic acid ((CF₃COOH) TFA) were purchased from Sigma-Aldrich and Alfa Aesar. 1 to 6 wt% chitosan solutions were prepared by dissolving chitosan in TFA in a sonicator bath (Branson Model 2510) at 55 °C for 90 minutes. After dissolution, the solutions were stirred for 15 minutes and filtered through mesh with 0.063 mm pore size to obtain homogeneous solutions and remove any undissolved particles. For electrospinning the solutions were added to 5 mL plastic syringe of 10 mm diameter with stainless steel needles of 0.2 and 0.8 mm diameters. The basic electrospinning setup (NANO-1A, Japan) used in this study is shown schematically in Fig.1. The syringe was placed in syringe pump and the needle was connected with high voltage supply, which could generate high voltage of up to 30 kV. In this study, the applied voltages were 14 kV, 18 kV and 22 kV, the flow rates were 0.4 mL/h and 0.8 mL/h and the distances between needle tip and collector were 100 mm and 140 mm. After electrospinning the nanofibers mats were removed from the aluminum foil (rolled on the rotating drum), dried in the vacuum oven at 60 °C and stored in the desiccator for further characterizations. The field emission-scanning electron microscopy (FE-SEM) micrographs of the Pt-coated electrospun chitosan nanofibers were taken using a Hitachi S-570 FE-SEM.

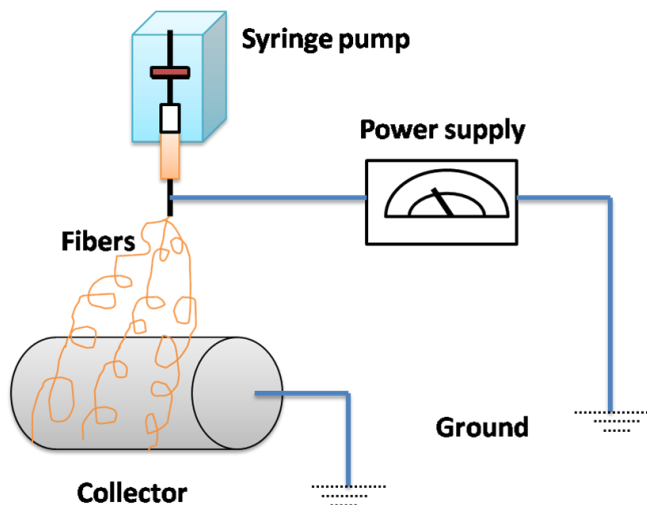


Fig. 1. Schematic representation of the electrospinning setup used in this study (NANO-1A).

3. RESULT AND DISCUSSION

Fig. 2. shows the FE-SEM micrographs of electrospun nanofibers. The micrographs showed partially electrospun nanofibers with beads and microspheres (Fig. a), decrease in the number and size of the beads (Fig. b, c), and beads elimination (Fig. e). The decrease in number and size of beads and microspheres (Fig. a-c) with increase in the voltage might be referred to the surface tension of polymer solutions; higher voltage overcame the surface tension of the polymer solution and initiated jet formation [11,12]. The slight increased bead size (Fig. d) with decrease in the needle tip to collector distance and increase in the voltage while keeping the flow rate and needle diameter constant might be attributed to the combine effect of small needle and rapid evaporation of the solvent at the needle tip. Solution flow rate and needle diameter are two essential factors, their optimization is also very important to maintain a stabilized Taylor cone during electrospinning [13]. Optimally, a lower solution flow rate, while maintaining the Taylor cone is more ideal since it permits time for solvent evaporation and produces better nanofibers. However, a solution flow rate below the threshold and small needle diameter for a given voltage might cause evaporation of the solvent at the needle tip, which not only block the needle tip but also prevent the formation of jet and resulting in no electrospinning or sometime in beaded nanofibers. Increasing the flow rate too much causes the nanofibers to be collected at the collector without sufficient evaporation of solvent, hence leading to flattened web-like appearance [14]. The observed bead free nanofiber morphology (Fig. e) when the needle diameter and flow rate was increased further supports our earlier arguments in support of Fig. d. Electrospun nanofibers were obtained (Fig. f) when the solution concentration was increased to 6 wt%, which might be attributed to the increased solution viscosity [15]. Increase in the polymer concentration has increased the viscosity of the solution to a critical value, therefore, eliminating the beads completely. When the polymer concentration is low, either many beads or microspheres appears in electrospun nanofibers, and the process become electrospinning when the concentration become low enough [15,8]. No electrospinning was observed for 6 wt% at 0.4 and 0.8 mL/h flow rate with 0.2 mm needle diameter, which is probably due to the blocking of the needle tip as result of the high viscosity of solution. Similarly the morphology of the nanofibers remain unchanged (Fig. g) when the needle tip to collector was increased to 140 mm.

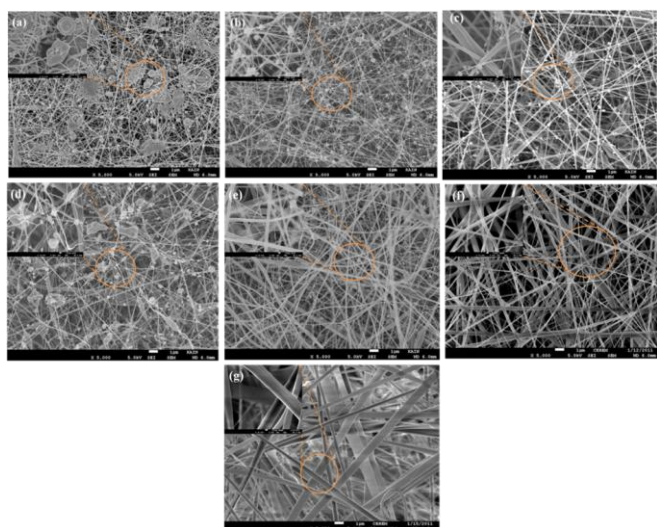


Fig. 2. FE-SEM micrographs of the electrospun nanofibers; (a) 4 wt%, flow rate = 0.4 mL/h, distance = 100 mm, needle diameter 0.2 mm, applied voltage = 14 kV, (b) 4 wt%, flow rate = 0.4 mL/h, distance = 100 mm, needle diameter = 0.2 mm, applied voltage = 18 kV, (c) 4 wt%, flow rate = 0.4 mL/h, distance = 140 mm, needle diameter = 0.2 mm applied voltage = 18 kV, (d) 4 wt%, flow rate = 0.4 mL/h, distance = 100 mm, needle diameter = 0.2 mm applied voltage = 22 kV, (e) 4 wt%, flow rate = 0.8 mL/h, distance = 100 mm, needle diameter = 0.8 mm, applied voltage = 22 kV, (f) 6 wt%, flow rate = 0.4 mL/h, distance = 100 mm, needle diameter = 0.8 mm, applied voltage = 22 kV and (g) 6 wt%, flow rate = 0.4 mL/h, distance = 140 mm, needle diameter = 0.8 mm, applied voltage = 22 kV.

4. CONCLUSIONS

Random bead free chitosan nanofibers were successfully prepared *via* electrospinning by controlling solution concentration, applied voltage, flow rate, distance between needle and collector and needle diameter. No electrospinning was observed for lower solution concentration i.e., 1-3 wt%, whereas a decrease in the number and size of the beads and microspheres, and bead free nanofibers were obtained when the concentration of solution was increased to 6 wt%, applied voltage to 22 kV, flow rate and needle diameter to 0.8 mL/h and 0.8 mm. Increase in the polymer concentration has increased the viscosity of the solution to a critical value, higher voltage overcame the surface tension of the polymer solution, changes in the flow rate, increased the flow rate above the threshold and change in needle diameter made the diameter large enough to avoid evaporation of the solvent at the needle tip and needle tip blocking, and thus has allowed the formation of jet. Flow rate, needle diameter and distance between the needle tip and collector effect was only observed 4 wt %, in all cases the number and size of bead decreased.

5. REFERENCES

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