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Fabrication and Characterization of Gelatin Nanofibers Dissolved in Concentrated Acetic Acid

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Abstract**—**Electrospinning is a simple, versatile and widely accepted technique to produce ultra-fine fibers ranging from nanometer to micron. Recently there has been great interest in developing this technique to produce nanofibers with novel properties and functionalities. The electrospinning field is extremely broad, and consequently there have been many useful reviews discussing various aspects from detailed fiber formation mechanism to the formation of nanofibers and to discussion on a wide range of applications. On the other hand, the focus of this study is quite narrow, highlighting electrospinning parameters. This work will briefly cover the solution and processing parameters (for instance; concentration, solvent type, voltage, flow rate, distance between the collector and the tip of the needle) impacting the morphological characteristics of nanofibers, such as diameter. In this paper, a comprehensive work would be presented on the research of producing nanofibers from natural polymer entitled Gelatin.

Keywords**—**Electro spinning, solution parameters, process parameters, natural fiber.

I. Introduction

ELECTROSPINING is a simple, convenient and unique process for producing ultra-fine fibers with a diameter range of 100 nm to 5 μm. More than 50 polymers have been successfully spun into fibers through this technique. When the diameter of polymer fiber materials decreases from micrometers to sub microns or nanometers, surface area, flexibility in surface, functionalities improve greatly, compared with any other known form of the material. It makes polymer nanofibers suitable for many important and interesting applications [1], [2]. Working parameters are very important to understand not only the nature of electrospinning but also the conversion of polymer solutions into nanofiber trough electrospinning. Each of those parameters can affect the fibers morphologies and by proper control of those parameters we will be able to fabricate electrospun fibers with the desired morphologies and diameters.

Most of the works reported on electrospinning involve the synthetic biodegradable polymer for dozens of applications in medicine, energy, transportation and electronic devices. In biomedical applications, synthetic biodegradable polymers, such as polyester regularly associated with poor biocompatibility and systemic or local reaction resulted from the acidic degradation products. Therefore, naturally occurring polymers such gelatin has been widely explored due to its biocompatibility, biodegradability, hydrophilic in nature and commercial availability at low cost [4].

Collagen is an aqueous polymer which is dissolvable in water. But unfortunately the gelatin/water system cannot be processed with electrospinning. Moreover, when dissolved in water at a temperature around or above 37 °C, gelatin becomes a kind of colloidal sol and hence without a special treatment (e.g., cross-linking) it is not suitable for tissue scaffold application [6]. Therefore, in this paper, the electrospinning of gelatin has been achieved by dissolving it in a non-toxic solvent acetic-acid. With this solvent, the gelatin solutions of mass concentration in 25 wt% have been successfully electrospun into ultra-fine fibers at room temperature. However, further lower concentrations were difficult to process. In this study, a concise introduction of solution and process parameters and their influence on synthetic nanofiber properties has been presented. The morphology of electrospun gelatin nanofibers was characterized using a scanning electron microscope (SEM). FTIR measurements were performed in a FTIR spectrometer to verify the composition of fibers for functional groups and determined whether acid has affected the gelatin structure or not. Thermal properties have been measured by DSC.

II. Processing

A. Solvent Selection

The solvent used to prepare the polymer solution has a predominant influence on its spinnability. Most research has been on the electrospinning of synthetic polymers, and limited to natural biopolymers, which are usually polyelectrolyte polymers. Gelatin can be easily dissolved in water but this solution is unable to be electrospun into ultra-fine fibers even under heated and non-gelation conditions. Unlike synthetic polymers that are generally nonionic and can be dissolved in organic solvents through nonionic interactions between solute and solvents, gelatin is a kind of polyelectrolyte polymer, which possesses many ionizable groups. Its amine and carboxylic functional groups can be ionized by acidic agents or hydrolyzed to carry positive or negative charges [5]. Gelatin is a biopolymer with strong polarity. There are very few high polarity solvents available for dissolving this biopolymer.

It is known that trifluoroethano and formic acid are good choices among other solvents. But it is worth mentioning that TFE is a toxic acid and formic acid will cause extreme break down gelatin structure [6]. Therefore, in this paper, the electrospinning of gelatin has been achieved by dissolving it in a non-toxic solvent.

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B. Electrospinning Process

The polymer solution has been loaded into a 10ml plastic syringe, and then it was placed in a syringe pump in order to control the flow rate. In electrospinning, an electric field draws a polymer solution from the tip of the capillary to a collector. A voltage is applied to the polymer, which causes a jet of the solution to be drawn toward a grounded collector, while the liquid jet is drawn to the collector, rapid evaporation solvent serves to solidify the fiber and prevent breakup [3].

III. Experimental

A. Materials

The materials which were used in this work include: (a) Gelatin of type B from bovine skin in powder form was purchased from Sigma Aldrich (Germany), (b) Acetic acid (CH_3COOH) and (d) deionized water figures.

B. Electrospinning

Gelatin solution 20% and 25% were prepared with concentrated acetic acid (90%) at room temperature. For instance, 20% w/v means that 2 g of gelatin powder was mixed with 10 ml of acetic acid solvent. An electrospinning unit from KATO TECH CO. was used. The syringe used had an 18-gauge needle (capillary diameter, 1.20 mm). The applied voltage was 15 kV and tip–to-collector distances and flow rate were fixed at 100 mm and 0.08 mm/min, respectively.

C. Characterization

The morphology of the electrospun mats were observed by a BAL-TEC SCD 005 SEM. Characterization of the chemical structure of the nanofiber samples was done by FTIR technique (Tensor 27, Bruker). The thermal properties of the electrospun have been measured by DSC.

IV. Result

A. Characterization /SEM Analysis

For evaluation, the effect of the dope concentration, the electrospinning was performed at various concentrations of 20 wt% and 25 wt% under the electric field of 30 Kv/cm and spinning distance of 10 cm. The morphological structures of the electrospun gelatin nanofibers were shown in Figs. 1-3. The SEM photographs showed that the gelatin nanofibers deposited randomly and their diameter were from several tens to a few hundred nanometers.

Fig. 1 shows the gelatin nanofibers obtained at different concentrations. Gelatin nanofibers, which had a diameter in a range of 35-250 nm were obtained at 25 wt% concentration, many beads, as well as droplets, were formed. In the case of low concentration, polymeric particles will be obtained and electrospray occurs instead of electrospinning. Owing to the low viscosity and high surface tensions of the solution, with a little higher concentration, a mixture of beads and fibers will be obtained. Finally, the smooth fibers can be attained by the appropriate concentration.

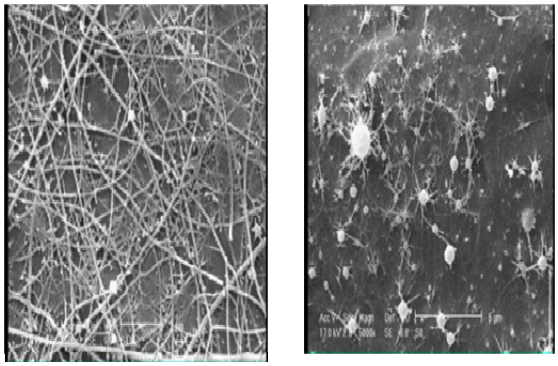

Fig. 1 SEM Images of gelatin electrospun fiber mats at a voltage of 30 kV, Collector distance 10 cm for different concentration, (A) 25 wt% (B) 20 wt%

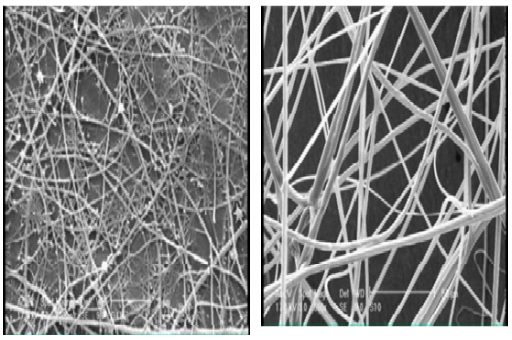

Fig. 2 SEM Images of gelatin electrospun fiber mats with 25% concentration. Collector distance 10cm for different concentration, (A) voltage 30kv (B) voltage 15 kV

Figs. 2 and 3 show that nanofibers with a diameter range of 35-250 nm were obtained at 15 kV voltage for both 20% and 25% concentration. Within the electospinning process, the applied voltage is a crucial factor. Only an applied voltage higher than the threshold voltage, charged jets to eject from the Taylor Cone (tip of the needle), can occur. However, the effect of the applied voltage on the diameter of electrospun fibers is a little controversial. As a result, a higher electric field produced beads and droplets more frequently because the spray of droplets, which were not charged enough to form a stable jet, occurred due to the very high electric field.

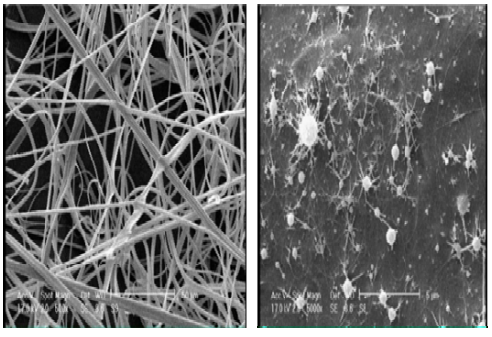

Fig. 3 SEM Images of gelatin electropsun fiber mats with 20% concentration, collector distance 10 cm for different concentration, (A) voltage 15 kV (B) voltage 30 kV

B. FTIR Spectroscopy

In order to investigate the possibility of the structural change of gelatin molecules in acetic acid, an FTIR spectroscopy analysis was examined in the range of 500-3500 $cm^{-1.}$ The electrospun gelatin had an amide (C=O stretch) at 1639-1700 cm^{-1}, amide II peak (N-H bend and C-H stretch) at 547-1639 cm^{-1} and amide A peak (N-H stretching vibration) at 3278 cm^{-1}, which are the distinguishing features of gelatin.

C. Differential Scanning Calorimetry

For investigating the thermal properties, DSC analysis has been done on the gelatin samples. Tg has been shown in 63 °C to 82 °C and the enthalpy is around -6.159 J/g.

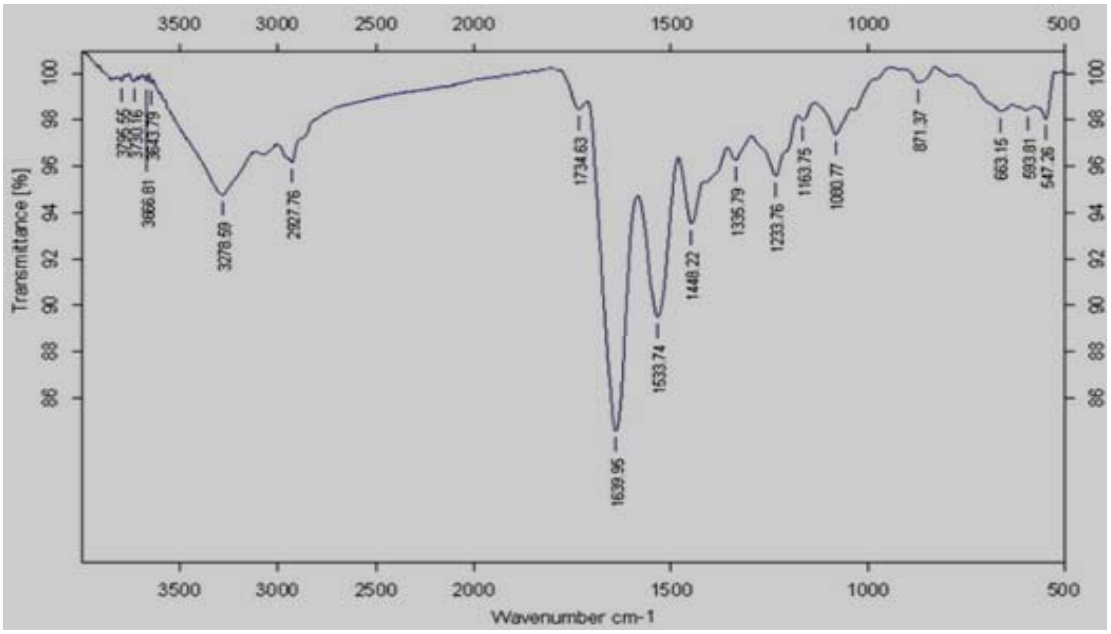

Fig. 4 FTIR spectra of gelatin nanofiber

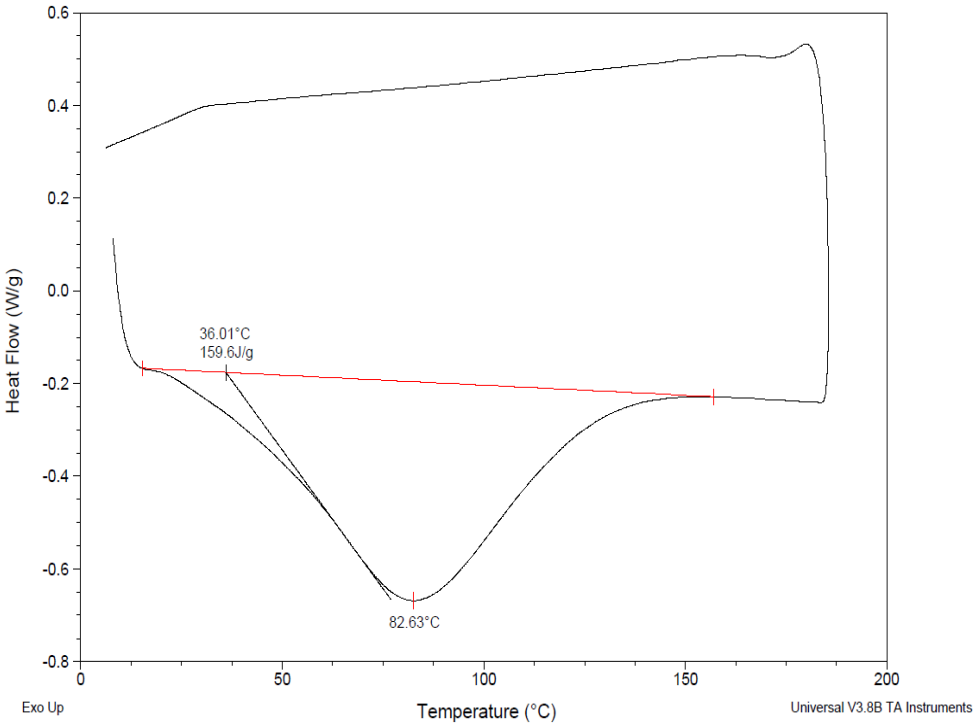

Fig. 5 DSC of gelatin nanofiber

V. Conclusion

Electrospinning of a natural biopolymer, gelatin was prepared with acetic acid as a solvent which has less effect on degradation, as it is weaker than other acids and also less toxic. Gelatin nanofibers were successfully prepared by the electrospinning of gelatin solutions with a gelatin concentration of 20% and 25% (w/v), which was prepared at a lower ratio compared with other researches. The electric field plays an important role in the electrospinning process, so it should be controlled properly for the preparation of gelatin nanofibers. Concentration, especially, was a major factor for the morphology by altering the size and bead formation. Uniform and very fine gelatin nanofibers, varying from 35 nm to 100 nm, could be produced by controlling the dope concentration under an electric field of 30 kV at a spinning distance of 10 cm. It is important to take all electrospinning parameters into consideration for the production of smooth and non-bead nanofibers.

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- **1st Conference on Textile Engineering with latest Methods in Related Industry,** Islamic Azad University Tehran Branch-Iran, 27 May, 2015, Review on Characterization & Application of Gelatin Polymer.
- **4th National Conference on Textile Engineering, Polymer** Islamic Azad University Yazd Branch- Iran, 17 Feburary,2016. Review on Electrospinning Parameters of gelatine Nanofibers.
- **-10th National Conference on Textile Engineering** Isfahan University of Technology –Iran, 26 April, 2016. The Effective Parameters of Electrospinning on Structure of Gelatin Nanofiber.
- **The Fiber Society Spring 2016 Conference, Textile Innovations-Opportunities and Challenges** Universite de Haute Alsace- Mulhouse, France 25 May, 2016. Fabrication and Characterization of Gelatin Nanofibers Dissolved in Concentrated Acetic Acid/Accepted.
- **13th International Conferences on N&N, Nanotexnology 2016,** Greece, 8July, 2016, Fabrication of Gelatin Nanofiber and the prospective application/Submitted, **Presenting author details,** Name: Kooshina, Full Name: Koosha, Contact number: +98-912-8054564, E-mail: koosha.ksh@gmail.com Category: (Oral presentation/ Poster presentation).