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# Tuning of Silk Fibroin Thin Films With Some Medicinal Herb Extract for Bio Medicinal Applications Using Electro Spinning Method

**Research Article** 

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# 1. Introduction

Silk as a typical fibrous protein is produced by a variety of insects including silkworm. Among the native silk proteins, the silkworm silk, mostly that of the domesticated Bombyxmori, has been used as high-quality textile fiber and suture for a long time. B. mori silk consists of two types of proteins, fibroin and sericin. Fibroin is the protein that forms the filament soft silkworm silk and gives silk its unique physical and chemical properties [1]. Sericins are the group of gummy proteins which bind the fibroin filaments. Silk fibroin (SF) can be used in various forms, such as gels, powders, fibers, or membranes, depending on application [2-3]. Besides its utility as a textile fiber, many researchers have recently investigated SF as one of candidate materials for biomedical applications because it has several useful properties including good biocompatibility, good oxygen and water vapor permeability, biodegradability, and minimal infiammatory reaction [4-5]. Practically, SF has been used in various fields such as cosmetics, medical materials for human health, and food additives. Recently, much attention has been paid to electrospinning process as a unique technique because it can produce polymer and processing conditions. In electrospinning, a high voltage is applied to create electrically charged jets of a polymer solution. These jets dry to form nanofibers, which are collected on a target as nonwovens. These nanofibers are of considerable interest for various kinds of applications, because they have several unique properties such as high specific surface area and high porosity. Examples are fiber membranes for filter applications [6] biomedical applications such as wound dressings and scaffolds for tissue engineering

Abstract: An electro spinning process was used to fabricate silk fibroin (SF) Nano fiber nonwovens for wound dressing applications. The electrospinning of regenerated SF was performed with formic acid as a spinning solvent. The morphology, conformational l structures of SF with herb extract nanofibers were investigated by scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDAX), Fourier transforms infrared spectroscopy (FT-IR), SEM micrograph showed that the electrospun SF nanofibers had an average diameter of 80nm and a distribution in diameter ranging from 30 to 120nm.

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[7-8] sensing applications [9-10], and fiber templates for the preparation of functional nanotubes [11-12]. The ultimate aim of this study is to develop wound dressings composed of the electrospun SF nanofibers. Up to date, some attempts to artificially spin silk protein fibers have been reported [13-14]. There has also been a preliminary report of electrospun spider silk nanofibers [15]. In the present paper, we describe how we have adapted the electro spinning process to fabricate an engineered matrix composed of SF nanofibers with extract of some medicinal herb for use in wound dressings. In addition, the chemical treatment to crystallize the as-spun SF nanofibers was conducted.

## 2. Experimental

Preparation of regenerated SF and electro spinning solution raw silk fibers were degummed twice with 0.5 % (w/w) NaHCO<sub>3</sub> solution at 100°C for 30 minutes and then rinsed with warm distilled water. Degummed silk (silk fibroin, SF) was dissolved in a ternary solvent system of CaCl<sub>2</sub>/CH<sub>3</sub>CH<sub>2</sub>OH/H<sub>2</sub>O (1/2/8 in mol ratio) at 70°C for 6 hours. After dialysis in distilled water for 3d, the SF solution was filtered and lyophilizedtoobtain the regenerated SF sponges. The SF solutions of 20wt% for electro spinning were prepared by dissolving the regenerated SF sponges in 98% formic acid for 3h. The three different solutions of the samples, they are a) Regenerated silk fibroin solution b) Medicinal herb extract c) Regenerated SF solution with medicinal herb extract.

#### 2.1. Electrospinning of Solution

In the electro spinning process, a high electric potential was applied to a droplet of SF solution at the tip (ID 0.495 mm) of a syringe needle, as shown in Figure 1. The nanofibers were collected on a target drum which was placed at a distance of  $\sim$ 7cm from the syringe tip for Regenerated silk fibroin solution, Medicinal herb extract and Regenerated SF solution with medicinal herb extract separately. A voltage of 15kV was applied to the collecting target by a high voltage power supply. The flow rate of polymer solution was 1.5mLh<sup>-1</sup>.

### 2.2. Characterization

Scanning electron microscope was used to investigate the macroscopic morphology surface texture for prepared fiber samples. FTIR spectra of electrospun SF fibers were obtained with a Travel IR in the spectral range of 4000-400 cm<sup>-1</sup>. EDAX experiments were performed on a different samples UV and XRD experiments also done.

# 3. Results and Discussion

The regenerated SF solution obtained by dissolving in formic acid was transparent. However, formic acid used for the preparation of SF solution is a moderate strong acid. Electro spinning general produces then non-wovens of randomly arranged ultrathin fibers with nanometer scale diameters.

### 3.1. Fourier Transform Infrared spectroscopy (FTIR)

FTIR has been often applied to study the molecular conformation of silk fibroin films. The FTIR spectra shown in Figure 1 used to determine the structural characteristics of two different samples they are a) Regenerated SF b) Regenerated SF solution mixed with medicinal herb extract. The secondary structure of B. mori silk fibroin consist of the major conformation including random Silk Fibroin. Conformation of silk fibroin shows strong absorption bands at 1665 (amide I),  $3428 \text{cm}^{-1}$  (amide II) and  $1570 \text{cm}^{-1}$  (amide III). Regenerated SF with Herb extract exhibits the spectral peaks of characteristics of  $\beta$ 

sheet of SF at  $1627 \text{cm}^{-1}$  and  $1700 \text{cm}^{-1}$  (amide1 structure) and  $1525 \text{cm}^{-1}$  (amide2 structure). FTIR of graph (b) slightly changed compare to (a) because of addition of Herb extract with silk fibroin.

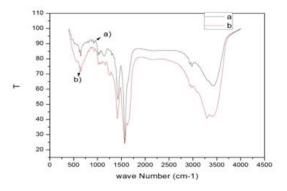


Figure 1: FT-IR spectra for a) Regenerated SF solution b) Herb extract c) SF with herb extract

#### 3.2. UV analysis

An investigation into the influence of UV-irradiation on a) Regenerated silk fibroin solution b) Medicinal herb extract c) Regenerated SF solution with medicinal herb extract was carried out using UV-Vis and fluorescence spectroscopy shown in Figure 2. It was found that the absorption peak appears at 250nm for both regenerated silk fibroin solution and regenerated SF solution with medicinal herb extract absorption peak appears at 340nm for medicinal herb extract of regenerated silk fibroin in solution increased during UV-irradiation of the sample, most notably between for regenerated silk fibroin solution and regenerated SF solution with medicinal herb extract and 400nm for herb extract. Moreover, after UV-irradiation a wide peak emerged between 290 and 340nm with maximum at about 305nm. The new peak suggests that new photoproducts are formed during UV-irradiation of regenerated silk fibroin. The absorbance peak was observed at 275nm for 305nm, at 480nm and at 601nm after excitation at. UV-irradiation caused fluorescence fading at 305nm and at 601nm. The increase of fluorescence was observed at 480nm, probably due to formation of new photoproducts. After excitation at 305nm the fluorescence of regenerated silk fibroin was observed at 340nm and at 400nm. UV-irradiation caused fluorescence fading at 305nm the fluorescence fading at 305nm the fluorescence fluorescence fading at 305nm the fluorescence f

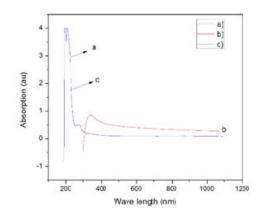
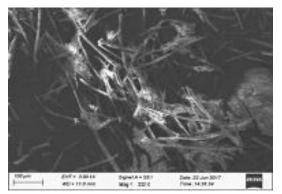


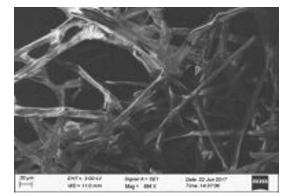
Figure 2: UV spectra for a) Regenerated SF solution b) Herb extract c) SF with herb extract

## 3.3. Scanning Electronic Microscope (SEM) Analysis

Figure shows SEM image of the a) Regenerated SF solution b) Regenerated SF solution mixed with Medicinal Herb extract. Both Nano fibers exhibits a circular cross section with a smooth surface. From the image analysis they have an average diameter of 80nm and their diameter ranged from 30nm to 120 nm. Image (b) shows that the smoothness of the fibers are improved and herb extract was filled between fibers.



(a) Regenerated SF nanofibers



(b) SF with medical herb extract

Figure 3:

#### 3.4. XRD Analysis

The SF and SF mixed with medicinal Herb extract Nano fibers showed no obvious  $2\theta$  peaks. Indicating that SF mixed with medicinal Herb extract conformation in spun state was mainly random coil. These results were consistent with those IR-Spectroscopy. This behavior can be explained by a higher surface area of non-wovens composed of SF with SF mixed with medicinal Herb extract nanofibers.

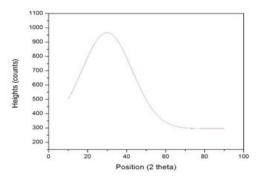


Figure 4: XRD graph for Regenerated SF with herb extract nanofibers

## 3.5. EDAX Analysis

Energy dispersive X-ray spectroscopy (EDAX/EDS) analysis of the Silk Fiber showing the presence of elements like Carbon, Oxygen. Sodium magnesium, sulfur chloride, Calcium and Iron in both a)Regenerated SF Nano fibers b)Medicinal Herb extract and c) SF with medicinal herb extract nanofibers. The results from domestic silk cocoon viz., Mulberry (Bombyxmori) has been documented. The ICP-MS results indicated the presence of C > O > Na > Mg > S > Cl > Fe, in a decreasing order of concentrations. The amount of the elements present in this SF reduces the smooth reeling of the silk thread.

# 4. Conclusion

The electro spinning of regenerated SF with herb extract was performed with formic acid as spinning solvent. The as spun SF nanofibers had an average diameter of 80nm and their diameter ranged from 20nm to 130nm.we investigated the influence of the additional medicinal herb extract on the secondary structure of Nano fibers by means of FTIR, EDAX, UV and XRD. With the help of electro spinning process we can fabricate an engineered matrix composed of SF with Herb Extract Nano fibers for wound dressing. EDAX shows the elements present in this SF reduces the smooth reeling of the silk thread. In future we can synthesis a smooth reeling SF by reducing the mineral content in the silk and is to develop a strong smooth reeling electrospun SF with herb extract nanofibers.

#### References

- D.Kalplan, W.A.Farmer and C.Viney, Silk polymers: Material science and Biotechnology, American chemical society, Washington DC., 6(1994), 370-717.
- M.Li, S.Lu, Z.Wu, K.Tan, N.Minoura and S.Kuga, Structure and properties of silk fibroin: Poly (vinyl alchol) gel, Int.
  J. Biol. Marcromol., 2(2002), 89-94.
- [3] S.Putthanarat, S.Zarkoob, J.Magoshi, J.A.Chen, R.K.Eby, M.Stone and W.W.Adams, Effect of processing temperature on the morphology of silk membrane, Polymer., 43(2002), 3405-3413.
- [4] H.Sakabe, H.Ito, T.Miyamoto, Y.Noishiki and W.S.Ha, Sen-I Gakkaishi, Miscibility, Structural characteristics, and thermal behavior of wet spun regenerated silk fibroin/nylon 6 blend filaments, 11(1989), 14-20.
- [5] W.H.Park, W.S.Ha, H.Ito, T.Miyamoto, H.Inagaki and Y.Noishiki, Fabrication and characterization of Nanofibrous scaffold developed by electrospinning, Fibers and Polymers., 14(2011), 317-325.
- [6] P.Gibson, H.Schreuder-Gibson and D.Rivin, Transport properties of porous membranes based on electrospun Nanofibers, Colloids surf., 187(2001), 469-481.
- [7] W.Li, C.T.Laurencin, E.J.Caterson, R.S.Tuan and F.K.Ko, Electrospun Nanofibrous structure: A novel scaffold for tissue engineering, Biomed. Mater. Res., 60(2002), 613-621.
- [8] E.D.Boland, G.L.Bowlin, D.G.Simpson and G.E.Wnek, *Electrospinning collagen and elastin: Preliminary vascular tissue engineering*, PMSE Preprints., 9(2001), 1422-1432.
- [9] S.A.Athreya and D.C.Martin, Sen. Actuators, Silk fibroin Nanofiber. Electro spinning properties, and structure, Polymer Journal, 35(2003), 185-190.
- [10] X.Wang, S.Lee, C.Drew, K.L.Senecal, J.Kumar and L.A.Samuelson, Proceedings of the ACS division of polymeric materials: Science and Engineering, PMSE Preprints., 112(2015), 22-26.
- [11] M.Bognitizki, H.Hou, M.Ishaque, T.Frese, M.Helling, C.Chwarte, A.Schaper, J.H.Wendorff and Grieiner, Polymer, Metal and Hybrid Nano and mesotubes by Coting Degradable Polymer template fibers (TUFT Process), Adv. Mater., 12(2000), 637-640.
- [12] R.A.Caruso, J.H.Schattka and A.Griener, Titanium Dioxide Tubes from Sol-Gel Coting of Electrospun polymer Fibers, Adv. Mater., 13(2001), 1577-1579.
- [13] R.L.Lock, Processing of bombay Mori Silk Using Ionic Liquids, U.S.Patent., 5(1993), 252-285.

- [14] K.A.Trabbic and P.Yager, Artificial Spinning of Spider silk, Macromolecules., 31(1998), 6733-6736.
- [15] Shahrzad Zarkoob, R.E.Eby, Darrel H.Reneker, Steven D.Hudson, Dale Ertley and Wade W Adams, Structure and morphology of electrospun silk nanofibers, Polymer., 45(2004), 3973-3977.