Electrospun Hybrid Films for Fast and Convenient Delivery of Active Herb Extracts

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Abstract: Herb medicines are popular for safe application due to being a source of natural herbs. However, how to deliver them in an efficacious and convenient manner poses a big challenge to researchers. In this study, a new concept is demonstrated that the electrospun polymer-based hybrid films can be a platform for promoting the delivery of a mixture of active herb extract, i.e., Lianhua Qingwen Keli (LQK), also a commercial traditional Chinese patent medicine. The LQK can be co-dissolved with the filament-forming polymeric polyvinylpyrrolidone K60 and a sweeter sucralose to prepare an electrospinnable solution. A handheld electrotweezing apparatus was explored to transfer the solution into solid nanofibers, i.e., the LQK-loaded medicated films. These films were demonstrated to be composed of linear nanofibers. A puncher was utilized to transfer the mat into circular membrane a diameter of 15 mm. Two self-created methods were developed for disclosing the dissolution performances of the electrospun mats. Both the water droplet experiments and the wet paper (mimic tongue) experiments verified that the hybrid films can rapidly disintegrate when they encounter water and release the loaded LQK in an immediate manner. Based on the reasonable selections of polymeric excipients, the present protocols pave a way for delivering many types of active herb extracts in an effective and convenient manner.

Keywords: medicated film; hybrid film; herb medicine; electrospinning; drug delivery; fast dissolution

1. Introduction

During the past half a century, polymers have acted as a backbone role to support the fast developments of pharmaceutics [1–8], particularly the dosage forms for drug delivery [9–16]. The numbers in which natural and synthetic polymers are introduced into this interdisciplinary field is always increasing [17–20]. Meanwhile, the techniques, the related methods and strategies for transferring a certain drug to a polymer-based medicated product are simultaneously expanded [21–26]. Thus, it is common sense that the joint efforts of advanced materials and advanced technologies have pushed the progress of effective, safe and convenient drug delivery.

As an important and also ancient section of medicine, Chinese herb medicine and the related Chinese patent medicine are modernized along different directions, such as effective extraction of the active ingredients from the herbs, combined applications with Western medicines and convenient and efficacious delivery of the multiple active ingredients contained in Chinese patent medicine [27,28]. Thus, it is not strange that nanomaterials and nanotechnologies have played important roles in modernizing traditional...
Chinese medicine [29,30]. Among different sorts of nanomaterials, polymer-based medicated films are one of the most popular ones [31–34], and show some advantages over other nanoproducts such as nanocrystals, nanolipids and inorganic nanocomposites in terms of processability, availability and feasibility [35–40].

Polymer-based medicated films can be presented in the form of round or concave particles, sheets, beads-on-a-string, dumbbells, tadpoles, rods and also linear fiber films [41–47]. Among these morphologies, nanofibers are a special one due to their one-dimensional format, i.e., diameter at a nano scale but with a size of length at macro scale [48–54]. To date, over 95% of the reported nanofibers are produced using electrospinning, which mainly present in a non-woven film [55]. As a popular electrohydrodynamic atomization (EHDA) method, electrospinning is similar to electrospaying in taking advantage of the easy interactions between working liquids and higher voltage electrostatic energy [56–62]. Their differences lie in the following: (1) the working fluids utilized in electrospinning often have a higher concentration and related viscosity than those exploited during electrospraying, and correspondingly (2) the products from electrospinning are fibers, whereas those from electrospraying are particles [63–67]. With either electrospinning or electrospaying, the functional ingredients can be co-dissolved with the polymeric matrices to experience the working processes for generating the desired polymer-based composites in a single step and a straightforward manner.

Often, a technique can be improved or developed along three directions, i.e., “practical, convenient and effective”. Electrospinning is now, on the one hand, moving forward from the single-fluid process [68–72] to coaxial [73–78], triaxial [79,80], side-by-side [81–84] and their combinations of multiple-fluid processes [85–88] for increasing its practicability and effectiveness of producing more kinds of complex nanostructures and the related multiple functional nanoproducts. On the other hand, electrospinning is increasing its easiness and feasibility for creating nanofibers on a large scale for possible commercial products [89,90]. Thus, the convenience of electrospinning and direct functional application of nanofibers comprises one of the important concerns in this field. Handheld electrospinning is one of the alternative solutions to this concern.

The Chinese patent medicine “Lianhua Qingwen Keli” (LQK) can be used for the treatment of influenza, particularly those patients with fever, cold aversion, muscle pain, nasal congestion and runny nose, cough, headache, dry throat and sore throat, red tongue and yellow or greasy coating. It can also be utilized for treating Coronavirus Disease 2019 (COVID-19) patients with fever, cough and fatigue [91]. However, LQK has a bitter taste, and has several adverse reactions, such as adverse gastrointestinal reactions, nausea, diarrhea, vomiting, abdominal pain, abdominal distension, dry mouth, rash, itching and dizziness [91]. Thus, new techniques for transferring LQK should be aimed to be more effective and compliant for drug delivery to the patients. Compared with traditional dosage forms, electrospun medicated nanofibers, often collected as a non-woven film, have a series of advantages for providing effective and convenient drug delivery. These advantages include small diameters of nanofibers, huge surface of the films and 3D web structures with high porosity [2,27,76]. Meanwhile, these advantageous properties can act synergistically to promote the fast dissolution of the loaded drug molecules [92,93].

In this work, a handheld electrospinning process is developed for a concept proof of the modernization of traditional Chinese medicine. With polyvinylpyrrolidone (PVP) as a filament-forming polymeric matrix and the commercial Chinese patent medicine product LQK as a model medicine composed of active herb extracts, a new type of medicated film in the form of electrospun films was generated using the handheld electrospinning process. The films were characterized using optical microscopy, scanning electron microscope (SEM) to detect their morphologies, X-ray diffraction (XRD) and Fourier-transform infrared (FTIR) to measure the components’ physical state, and two methods were developed to evaluate the functional performances of the prepared LQK-loaded hybrid films. The results demonstrated that the electrospun hybrid films can be a potential candidate for the fast and convenient oral delivery of active herb extracts.
2. Experimental

2.1. Materials

Polyvinylpyrrolidone K60 (PVP K60, Mw = 360,000, CAS No. 84057-81-8) and sucralose (CAS No. 56038-13-2) were purchased from Sigma-Aldrich Corp. (Shanghai, China). Lianhua Qingwen Keli (LQK, brown particles) was commercial products from a local pharmacy (Lao-Bai-Xing Big Pharmacy, Shanghai, China). Water (both for preparing electrospinnable fluid and for fast disintegrating experiments) was double distilled just before use.

2.2. Preparation of Electrospun Films

The working fluid was a blending solution of PVP K60, LQK and sucralose. The concentrations (g/g) were 10%, 5% and 1% in distilled water, respectively.

HHE-1 handheld electrospinning apparatus (Qingdao Junada Technology Co., Ltd., Qingdao, China) was exploited for the preparation of electrospun films. The apparatus is easy to operate, safe and reliable, and has high integrity. It realizes the in situ real-time spinning of electrospun nanofibers, and at the same time strengthens the design of electrostatic safety protection and ergonomics.

A cardboard wrapped with aluminum foil was used as a plate fiber collector. The distance between the nozzle of spinneret and the collector was about 15 cm. The resultant nanofiber mats were stored in a vacuum dryer (DZF6090, Shanghai Precision Instrument Co., Ltd., Shanghai, China) to reach a constant weight. The ambient temperature and relative humidity were 21 ± 3 °C and 43 ± 5%, respectively.

2.3. Characterizations of Nanofibers

The resultant nanofibers’ morphologies were assessed using a field-emission scanning electron microscope (FESEM, Hitachi, Tokyo, Japan). A little patch was cut from the electrospun nanofiber membranes and was adhered on the conductive tape on a sample stage. Later, the samples were coated with a thin layer of Pt through a sputter for 1.5 min under a N2 atmosphere. Additionally, an optical microscope (WMS-3590, Shanghai Wu-Mo Optical Instrument Corp., Shanghai, China) was utilized for the assessment of resultant nanofibers. The sampling was realized by putting a slide glass under the handheld apparatus for several minutes.

X-ray patterns of the LQK, PVP, sucralose and their electrospun hybrid nanofibers were achieved using a high-resolution X-ray diffraction system (XRD, Bruker-AXS, Karlsruhe, Germany), in which CuKα is explored as the radiation source. The experimental conditions include 45 kV, 40 mA, 1.5 Å and a θ range from 10° to 60°.

Fourier-transform infrared analyses were conducted using a spectrophotometer (FTIR, Spectrum 100, Billerica, MA, USA). All spectra were obtained at room temperature within a wavenumber range of 600–4000 cm\(^{-1}\) with acquisition of 16 scans and 2 cm\(^{-1}\) resolution.

2.4. Functional Performances

Two self-created methods were explored to evaluate the fast-disintegrating and fast-dissolving processes of the prepared orodispersible films. A puncher was exploited to transfer the electrospun mats into the orodispersible films. One method was to drip a droplet of water on the nanofiber mats collected on a glass slide. The other method was to place a piece of electrospun hybrid film on wet paper. All these processes were recorded using a digital camera (PowerShot SX50HS, Canon, Tokyo, Japan) [76,94].

3. Results and Discussion

3.1. Electrospinning Is Explored to Transfer LQK into Nanofiber Mats

In the market, LQK has two traditional dosage forms, one is in particles and the other is in capsules. The active ingredients are extracted from herbs including forsythia, honey-suckle, prepared ephedra, fried bitter almond, gypsum, isatidis root, cotton horse rhizome, houttuynia, patchouli, rhubarb, rhodiola, menthol and licorice [91]. The LQK is brownish yellow to brown particles, with gas that is slightly fragrant and tastes slightly bitter. In this
work, a strategy is developed for transferring these particles into medicated hybrid films, which is exhibited in Figure 1. A filament-forming polymer—PVP, and a common sweeter sucralose were co-dissolved with LQK into water to prepare the electrospinnable working fluids. PVP is a well-known filament-forming polymeric matrix and also a useful solubility enhancer of a wide variety of poorly water-soluble drugs. Thus, the combinations of PVP, sucralose and LQK are anticipated to promote the fast dissolution of active ingredients from the herb extracts and meanwhile change them to a favorable taste for the patients. Accordingly, the handheld electrospinning is a green process healthy to the environment and also benefits the final products, thanks to being free of any organic solvents. Finally, the resultant nanofiber mats could be cut into circular films for easy oral administration.

![Figure 1](image-url) A scheme showing the procedure from the (1) reasonable selection of matched raw materials, to (2) facile implementation of a green preparation using handheld electrospinning, and to (3) systematic characterizations of the nanofibers and the converted medicated membranes.

Electrospinning is a special micro- and nanofiber manufacturing process in which a polymer solution is jet-spun in a strong electric field [68–72], just as its EHDA brother method, electrospraying [95–97]. Under the action of the applied high voltage, the droplets at the nozzle are changed from a spherical to a conical shape (i.e., the famous Taylor cone), and later split and stretched from the tip of the cone to remove the loaded solvents for creating solid micro- and nanodiameter fibers. In the HHE-1 handheld electrospinning equipment, two cells are utilized to provide the high voltage (typically $0 \pm 10 \pm 1$ kV, and the rated current is often within 90 mA). Its whole weight is only 133 g, very slight for application. The designed syringe size is 5 mL. The suitable working ambient conditions are similar to other electrospinning systems, i.e., a temperature range of 0–40 °C and a working environment relative humidity of smaller than 80%.

Shown in Figure 2a is the working fluid, which has a brown color. The HHE-1 handheld electrospinning instrument is flexible and easy to operate. A syringe (5 mL) was perfused with the prepared working fluid, and then put into the body of the HHE-1 handheld instrument, a stainless-steel needle (20 G × 38 mm length) was connected with the syringe, and later, the button was pushed to start the machine. When the red indicator light was always on (Figure 2b), the operator held the instrument to keep a certain distance (here 15 cm) from the receiving aluminum foil. The operator needed to keep his press continuously on the button of the electrospinning apparatus, and slowly push the pushing rod of the syringe with the thumb. After adjusting the distance between the apparatus and the receiving foil and the speed with which the thumb pushed the pushing rod to achieve the best effect, the nanofiber mats could be collected. For safe operation, it is strictly forbidden to touch the needle and the directional cover when the instrument is powered.
on, so as to avoid the danger of applied high voltage. In the present study, a typical electrospinning process and the typical Taylor cone are given in Figure 2c,d, respectively.

3.2. The Morphologies of the LQK-Loaded Nanofibers and Their Transferring to Orodispersible Films

The morphologies of the electrospun nanofibers detected using different microscopes are shown in Figure 3. Both the OM (Figure 3a) and SEM images (Figure 3b,c) indicate that these nanofibers presented in a straight linear format, with few discerned beads-on-a-string or spindles-on-a-string phenomena. They have an estimated diameter of 860 ± 130 nm (Figure 3d). As a linear polymer, PVP has fine filament-forming properties. Meanwhile, PVP has a fine solubility in water and a wide variety of organic solvents such as ethanol, methanol and acetone. Thus, PVP, being authorized by the Food and Drug Administration (FDA) for biomedical applications, is frequently explored for applications in many sorts of pharmaceutical dosage forms such as pellets, tablets and capsules. Here, the LQK-loaded films demonstrate an additional manner for modernizing traditional Chinese patent medicines using PVP as a polymeric matrix.

Electrospun medicated nanofibers are mainly intermediate dosage forms, which can be transferred into different final dosage forms for various administration applications [93].
Here, a puncher was used to transfer the electrospun nanofiber mats into orodispersible films. The digital pictures in Figure 4 show the whole process. The stainless-steel puncher has a circle cut diameter of 15 mm. After pressing on the electrospun nanofiber mats with force (Figure 4a), a circle with a diameter of 15 mm (Figure 4b) can be cut off for administration applications. The weight of each film is 7.8 ± 1.4 mg (n = 10), and thus containing 2.5 ± 0.4 mg LQK.

**Figure 4.** Conversion of the electrospun nanofiber mats to orodispersible films: (a) puncher is exploited to cut membrane; (b) the diameter of the cut circle.

### 3.3. Physical State of the Components Loaded into the Films

The crystal property of the raw materials and the electrospun nanofibers can be detected using the XRD patterns. Shown in Figure 5, it is clear that sucralose presents in a crystalline format and the granular mixtures of LQK contain some crystal active ingredients. PVP is a well-known amorphous linear polymer, which has fine electrospinnability and is able to prevent the crystallization of many drugs. As anticipated, the electrospun films were also in an amorphous state. The crystalline materials in LQK and sucralose lost the crystal state and were converted into the hybrid films amorphously during the electrospinning processes, which could benefit the fast dissolution of LQK’s ingredients due to no lattice energy needing to be overcome for dissolution.

**Figure 5.** XRD patterns of the raw materials sucralose, LQK, PVP and their electrospun films.

Shown in Figure 6 are spectra of the raw materials (LQK, sucralose and PVP) and their electrospun nanofibers. A physical mixture of the raw materials was detected for comparison. LQK is a mixture containing a series of active herb extracts. It has characteristic peaks at 1657, 1471, 977 and 864 cm\(^{-1}\). These peaks can also be found in the spectra of the mixture. However, in the spectra of electrospun films, they disappeared. These phenomena...
suggest that the favorite secondary interactions should happen between those active herb extracts and PVP, which maybe include hydrogen bonding, hydrophobic interaction and electrostatic interactions [28,46,76]. This is favorable, on the one hand, for the fast dissolution of LQK. On the other hand, it is desirable for the stable storage of the films.

3.4. The Fast-Disintegrating Performances of the LQK-Loaded Films

As shown in Figure 7, when a drop of water was dripped onto the slide glass covered with the electrospun nanofiber mats, the logo of “School of Materials and Chemistry” appeared gradually but quickly. The time period from “1” to “9” was just 6 s, suggesting the easy and rapid dissolution of PVP, sucralose and the loaded active herb extracts in LQK.

Two papers were superposed and placed on a Petri dish. After wetting with some water to mimic a tongue, a piece of the cut film was placed on the wet paper. A camera was used to record the whole process. The dissolution and passive diffusion processes were shown in Figure 8. The time from “1” to “6”, which is a water-absorbing and gelling process, took only 4.2 ± 0.6 s. After absorbing water, the opaque and slight yellow color of the membrane was gradually turned to a yellow-brown color and transparent. Apparently, the
hygroscopicity and hydrophilicity of the PVP matrix, the small diameter of nanofibers, the amorphous state of the components disclosed by XRD patterns and the three-dimensional web structure of the nanofiber films acted together to promote the gelling processes. Later, the yellow-brown color was gradually weakened, as shown from “7” to “9”. This is a passive diffusion process, lasting a relatively longer time period of 91.7 ± 11.4 s. If extra stirring was added (such as mimicking the tongue movement), the diffusion and transportation processes should be still very quick. Incidentally, the films need to be stored in an environment with a low humidity due to the hygroscopicity of the polymeric carrier, i.e., PVP, which is also a common issue for numerous traditional dosage forms.

![Figure 8. Fast-disintegrating experiments of an orodispersible film on the interfacial tongue.](image)

4. Conclusions and Perspectives

With PVP K60 as a drug carrier and also a filament-forming polymeric matrix, a handheld electrospinning apparatus was exploited to prepare hybrid films containing active herb extracts in a traditional Chinese patent medicine, LQK. A sweeter sucralose was co-loaded into the working fluids, which can be explored to cover up the bitter taste of the original commercial LQK products. The electrospun LQK-loaded films were demonstrated to be linear nanofibers, which had a diameter of 860 ± 140 nm. A puncher was utilized to transfer the electrospun mats into circular films. XRD and FTIR experiments demonstrated that the green electrospinning process successfully converted the crystalline herb components and sucralose into an amorphous state due to the secondary interactions. Two self-created experiments verified that the prepared orodispersible films showed the desired functional performance in promoting the fast disintegration and dissolution of LQK, which can increase the convenience for the patients. The sweet taste and the convenience of administration should improve the patients’ compliance of LQK.

The present study pioneers a concept for the modernization of traditional Chinese medicines and for effectively delivering herb extracts. Often these medicines and herb extracts are known as safe for the human body due to a natural source of herbs. However, their effectiveness and convenience need to be improved. The electrospun polymeric nanocomposite membrane can be a useful strategy for transferring them into a more efficacious dosage form. Particularly, the liquid and paste dosage forms are very frequently in traditional Chinese patent medicines due to an extraction process of the active ingredients from the herbs. These dosage forms can be solidified into solid nanofibers for easy shipping and transportation, and also a longer time period of the stability of active ingredients. Additionally, the combined therapy of traditional Chinese medicine and Western medicine...
is popular. The present protocols can be further extended to prepare dosage forms that contain both the Chinese patent medicines and bioactive chemical little molecules in future.

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**Abbreviations**

Coronavirus Disease 2019: COVID-19; Lianhua Qingwen Keli: LQK; Electrohydrodynamic atomization: EHDA; Polyvinylpyrrolidone K60: PVP K 60; Scanning electron microscope: SEM; Field-emission scanning electron microscope: FESEM; X-ray diffraction: XRD; Fourier transform infrared: FTIR; Food and Drug Administration: FDA.

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