Nanofiber Mask Fabrication by Electrospun and Its Application

Wen-Ta Su* and Ming-Shiuan Hsu
Department of Chemical Engineering and Biotechnology, National Taipei University of Technology, Taipei, Taiwan
1 Sec. 3, Chung-Hsiao E. Rd, Taipei 10608, Taiwan
*Corresponding author. Tel.: +886-2-27712171 ext. 2554; Fax: +886-2-27317117
E-mail: f10549@mail.ntut.edu.tw

Abstract. Electrospinning technology is a novel nano-process technology in recent years. It can directly and quickly spray polymer into nano scale fibers, improve the specific surface area, porosity and microporous structure of materials, and extend materials to different applications. Blend PVA/gelatin/magnesium ascorbyl phosphate/tranexamic acid solution, and spin the fibrous mask of 600~800 nm by electrospun spinning machine. Analyze the composition of the fibrous mask, mechanical strength, swelling ratio and nutrients release rate. Finally, evaluate the effect of nanofiber mask by human body test. This study provided a quality nanofiber mask that will be commercially available in the future.

1. Introduction
Nanofibers, which are composed of ultrafine and continuous fibers, have recently attracted attention in regenerative medicine engineering and cosmetic industry because of their high porosity, variable pore-size distribution, high surface-to-volume ratio, and capacity to perfectly attach the face for cosmetic engineering [1,2]. Electrospinning uses an electric field to convert a polymer solution into continuous polymer fibers, and can be applied to fabricate fibrous membrane that mimic the structure of the commercial mask. The resulting fibers with diameters ranging from microns to a few nanometers have a nonwoven structure, which can more attach to the skin on the face.

Polyvinyl alcohol (PVA) is a water-soluble, biodegradable and synthetic polymer prepared from polyvinyl acetates by replacement of the acetate groups with hydroxyl groups. It is identified by the formula [CH₂CH(OH)]ₙ. PVA is a highly biocompatible, hydrophilic, non-toxic, biologically stable polymer that possesses excellent film-forming, emulsifying, adhesive, and flexible mechanical properties [3]. Gelatin is a mixture of peptides and proteins produced by partial hydrolysis of collagen extracted from the skin, bones, and connective tissues of animals. Gelatin is a biodegradable natural polymer material and can be used as biological glue, wound dressing or drug carrier after cross-linking modification on biomedical materials [4]. Magnesium ascorbyl phosphate and tranexamic acid are whitening ingredients, which can decrease the activity of casein enzyme, inhibit melanin production, and achieve whitening effect.

In this study, we investigate the possible effect of nanofiber mask by electrospun and then blended with whitening ingredients to test the whitening effect of the actual beauty face.
2. Materials and Methods

2.1. Nano Fiber Mask Fabrication
Take 1 g of water-soluble PVA into 5 mL of deionized water, and then continue heating by stirring for 1 hour at 80-85 °C using a water-blocking heating method. Weigh 0.5 g of gelatin into 5 mL of deionized water, and then continue to shake with an ultrasonic oscillator for 1 hour to completely dissolve the gelatin particles. Two solutions well mixed for 1 day, and then added 0.1 g magnesium ascorbyl phosphate, 0.45 g tranexamic acid, 0.2 mL glycerin and 0.9 mL propylene glycol were blended. The homogeneous solution was loaded into a 10 mL syringe connected to an 18-gauge needle with a distance of 15-25 cm between the tip and the aluminum foil collector, and the device was placed into the syringe pump of an electrospinning system. Optimization the fabrication of nanofiber mask by stimulating solution flow rate, voltage of power, working distance and rotational speed of collector. Electrospinning was performed for 8h to obtain nanofibrous mask.

2.2. Physical Properties of Nanofiber Mask
The appearance and the thickness of the PVA/gelatin nanofiber mask were observed by scanning electron microscope (SEM), the fibers were dehydrated in sequentially increasing ethanol solutions to 100 vol % and then dried using a CO₂ critical-point dryer (Ted Pella Inc., USA). The specimens were coated with a thin layer of gold (Cressington 108 manual, Ted Pella, Inc., USA) and then examined under an SEM (FEI Quanta 200, Thermo Fisher Scientific, USA). The diameter of the fabricated fibers was quantified with image analysis software (Image J, NIH), and the fiber average diameter was obtained by measuring the diameters of 100 randomly selected fibers.

2.3. Swelling ratio and Moisture Content of Nanofiber Mask
To evaluate the water absorption and moisturizing properties of nanofiber mask, takes about 0.5 g of PVA/gelatin nanofiber mask and dried in an oven at 50 °C and weigh (dry weight) until the weight shows a stable value. Then mask was immersed in deionized water at room temperature for 30 min. The water on the surface of the mask was wiped out and weighed (wet weight). Swelling ratio (SR) and moisture content (MC) are as follows:

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SR = \frac{W_{\text{wet}} - W_{\text{dry}}}{W_{\text{dry}}} \times 100\%
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MC = \frac{W_{\text{wet}} - W_{\text{dry}}}{W_{\text{wet}}} \times 100\%
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2.4. Composition and mechanical property assay
The composition of nanofiber mask was analyzed using Fourier transform infrared (FTIR, HORIBA FT-720) spectroscopy. Mechanical characterization measurement was performed on the nanofiber mask using a universal testing machine (PT-003, Kotsao, Taiwan). The tested sample was cut into a thin strip, and a tensile test was conducted at a crosshead speed of 50 mm/min at room temperature.

2.5. Nutrients Releasing Assay
1 cm × 1 cm nanofiber mask immersed into 20 mL, 1% PBS solution, and measured the absorbance at 210 nm (tranexamic acid) and 254 nm (magnesium ascorbyl phosphate) by UV-spectroscopy (Biomate 3, Thermo) for 1, 5, 10, 15, 30, 40 and 60 min, respectively.

2.6. Mask Attachment Test
The PVA/gelatin nanofiber mask was cut to a size of 3 cm × 1 cm, and then torn off from the aluminum foil, and then the right cheek area was wet with distilled water, and the nanofiber mask was slowly applied against the face for 15-20 minutes, imitating the general use of commercial masks, twice a week for one month.

3. Results
The electrospinning process can change the type of the collection plate, the collection plate rotation speed, the feed flow rate, the type of solution, solution concentration, voltage, working distance, injection needle size and other variables can produce different fiber types. These processes change the
traditional inherent 2D plane form, and retain or improve the advantages of nanometer fiber itself such as small diameter, high porosity and network-like structure, and increase the diversity of nanometer fiber form. Optimized electrospinning conditions: a voltage of 20 kV, a flow rate of 1.2 mL/h, and a working distance of 25 cm. Nanofiber masks can be successfully fabricated. The average diameter of the fiber mask will be between 600 and 850 nm.

3.1. Morphology of Nanofiber Mask
A preliminary comparison of the PVA/gelatin/nutrients nanofiber mask with a commercial mask was performed by visual inspection and SEM photographing, respectively. It can be found that the appearance of the nanofiber mask is not much different from the commercial mask, except that the nanofiber mask is thicker, as shown in Fig. 1. Comparing the distribution of fiber diameters between the two, the average diameter of the nanofiber mask is between 600 and 850 nm (Fig. 1e), and the diameter of commercial mask is between 15 and 30 μm (Fig. 1a). Therefore, when attaching to the skin, because the nanofiber mask has a large specific surface area, it releases the nutrients faster per unit time, so it has obvious advantages over the non-woven carrier.

![SEM micrograph of commercial mask (a, c) and nanofiber mask (b, d) and the diameter distribution of nanofiber mask (e)](image)

**Figure 1.** SEM micrograph of commercial mask (a, c) and nanofiber mask (b, d) and the diameter distribution of nanofiber mask (e)

3.2. Swelling Ratio and Moisture Content of Nanofiber Mask
The nanofiber mask fabricated by electrospinning containing two kinds of hydrophilic polymers, water-soluble PVA and gelatin, so this nanofiber mask has good swelling ratio 927% and high moisture content 90%, as shown in Fig. 2. Compared with the commercial mask, although the performance of the swelling degree is slightly inferior to the commercial mask of 1196%, the moisture content can reach more than 90%.
3.3. Composition and Mechanical Property of Nanofiber Mask

Figure 3a shows the characteristic functional groups of PVA have absorption peaks near (-OH) at 3000 ~ 3600 cm\(^{-1}\), (-CH) at 2932 cm\(^{-1}\), (-CH\(_2\)) at 1455, 1336 and 1240 cm\(^{-1}\) [5]. C = C and C = O of gelatin show absorption peaks at 1450 ~ 1688 cm\(^{-1}\) and 1680 ~ 1850 cm\(^{-1}\), respectively; -COO' characteristic functional groups representing tranexamic acid are located at 1630 and 1412 cm\(^{-1}\). It is covered because the absorption peak of carbonyl (C = O) is too strong, but it can be indirectly proved to contain this component by -NH\(_2\) stretching vibration (2700 ~ 3100 cm\(^{-1}\)) and -NH\(_2\) bending vibration (1510 cm\(^{-1}\)), and the addition of tranexamic acid, the wave peak will have a shift phenomenon. The characteristic functional groups of magnesium ascorbyl phosphate are the same as those of PVA and gelatin, and the characteristic peaks of -OH and C = O can be found at 3000 ~ 3600 cm\(^{-1}\) and 1716 cm\(^{-1}\), respectively.

PVA containing 1%, 3%, and 5% gelatin is lower in stress tolerance than pure PVA, and the maximum stress has dropped from about 6 MPa to 4 MPa, 2.3 MPa, and 1.8 MPa, respectively. However, with the increase of the amount of gelatin, the strain performance of the film is improved (Fig. 3b). In general, the mask carrier mainly focuses on the degree of deformation and the ability to carry nutrients, and this result can clearly show that the PVA/gelatin/nutrients material has good resistance to strain.

Figure 2. The swelling ratio and the moisture content of nanofiber mask for different composition.

Figure 3. The FTIR of the PVA/gelatin/nutrients nanofiber mask (a), and the stress-strain curve of different content of gelatin in 10% PVA (b).
3.4. Nutrients Releasing

Figure 4 shows that within 10 to 15 minutes, the release curve of magnesium ascorbyl phosphate and tranexamic acid has a sudden release before reaching stability, which is consistent with the zero-order drug release system proposed by Xiao and Huang [6]. This phenomenon resulted in the nutrients are randomly distributed on the nanofibers, so the nutrients will be quickly released into the PBS and a burst release will occur.

![Figure 4](image1.png)

**Figure 4.** The releasing rate of magnesium ascorbyl phosphate (left) and tranexamic acid (right) in PBS

3.5 Mask Attachment Test

The actual comparison before and after the use nanofiber mask and the subjective feelings of the subjects were comprehensively analyzed. All subjects were objectively analyzed on the skin of face; the results shown in Figure 5. The skin condition has improved significantly with the increase in the number of uses. Due to the large specific surface area of the nanofiber mask, the degree of moisturization is no different from that of general commercial masks and the high degree of serviceability makes it easier to release the nutrients to the skin surface. After continuous use, the phenomenon of easy oiling on the face, sagging skin, pore expansion, and melanin deposition will all improve.

The nutrients are without any preservatives and irritating additives, so there will be no redness, itching and allergies during use. After the test, all the participants agreed that the nanofiber mask was rated as excellent in terms of whitening, moisturizing and conformability.

![Figure 5](image2.png)

**Figure 5.** The objective feeling after using the nanofiber mask for subjects
4. Discussion
Electrospinning technology is used to fabricate PVA/gelatin/nutrients nanofiber masks. Under different operating parameters, the effects of fiber diameter changes and mask appearance found the optimal conditions for electrostatic spinning. With a concentration of 5% gelatin at a voltage of 20 kV, a flow rate of 1.2 mL/hr, and a working distance of 25 cm, a nanofiber mask with a diameter of 600 to 850 nm can be successfully prepared. It is analyzed that the maximum tensile strength is 11.2% higher than the commercial mask; the resistance to external force deformation is also 105% higher than the commercial mask. FTIR analysis identified the functional groups, and found that there was no significant chemical shift in the functional groups, and the signals of tranexamic acid and magnesium ascorbyl phosphate apparently appeared on the fibers, proving that the additives successfully bind to the nanofibers. In terms of nutrients release results, the main whitening ingredients, tranexamic acid and magnesium ascorbyl phosphate, can accumulate release rates of 22% and 60%, respectively, within 30 minutes, which indeed shows the effect of nutrients release. Nanofiber mask is different from the common non-woven, cotton cloth and other mask carriers on the market. It has an ultra-high specific surface area, nano size, moisturizing ability, and the most comfortable.

5. Conclusions
This study demonstrated that electrospinning technology can be used to fabricate a multifunctional mask, which can improve the specific surface area and attachability of the mask. Nanofiber mask present nano scale and significantly improves stress and strain performance compared to the commercial mask. The used effect is excellent, so nanofiber mask can be developed and manufactured towards commercial products in the future.

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7. Reference
[1] Wen-Ta Su, Pai-Shuen Wu, Te-Yang Huang, Osteogenic differentiation of stem cells from human exfoliated deciduous teeth on poly(ε-caprolactone) nanofibers containing strontium phosphate. Materials Science and Engineering: C 2015; 46,427-34.
[2] Wen-Ta Su, Yi-Jie Liu, Te-Yang Huang. Nanofibers promote HepG2 aggregate formation and cellular function. Genetics and Molecular Research 201615 (3); gmr.15038993.
[3] Te-Yang Huang, Guo-Shou Wang, Chia-Chen Tseng, Wen-Ta Su, Epidermal cells differentiated from stem cells from human exfoliated deciduous teeth and seeded onto polyvinyl alcohol/silk fibroin nanofiber dressings accelerate wound repair. Materials Science and Engineering: C ( 2019, in press)
[4] Kodjo Boady Djagnya, Zhang Wang, Shiyiing Xu , Gelatin: A Valuable Protein for Food and Pharmaceutical Industries: Review. Critical Reviews in Food Science and Nutrition. 2010; 41 (6): 481-92.
[5] Kim KO, Y, Wei Kai, Kim BS, Kim IS, Cells attachment property of PVA hydrogel nanofibers incorporating hyaluronic acid for tissue engineering. Journal of Biomaterials and Nanobiotechnology, 2011, 2, 353-60.
[6] Huang X., Brazel CS, On the importance and mechanisms of burst release in matrix-controlled drug delivery systems, Journal of Controlled Release, 2001; 73,121-36.
[7] Su WT, Shih YA, Ko CS. Effect of chitosan conduit under a dynamic culture on the proliferation and neural differentiation of human exfoliated deciduous teeth stem cells. J Tissue Eng Reg Med 2016; 10,507-17.