ACS OMEGA

Open Access

http://pubs.acs.org/journal/acsodf

Article

Green and Sustainable Electrospun Poly(vinyl alcohol)/Eggshell Nanofiber Membrane with Lemon-Honey for Facial Mask Development

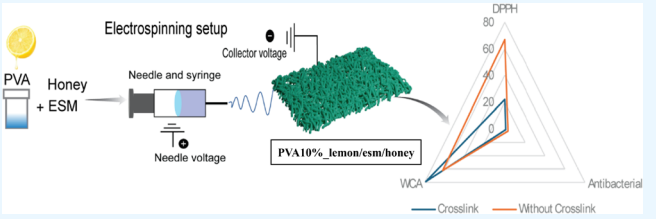
Maira Khalid, Muhammad Farooq,* Muhammad Adnan, Shoki Kobe, Gopiraman Mayakrishnan, and Ick Soo Kim*

Cite This: *ACS Omega* 2025, 10, 12972−12982

Read Online

ACCESS | Metrics & More | Article Recommendations | Supporting Information

ABSTRACT: Facial masks contain additives such as thickeners and preservatives that have adverse effects on the skin, and there is growing demand for organic products. Inspired by this, we developed a poly(vinyl alcohol) (PVA) nanofibrous facial mask that contains all-natural ingredients such as honey and an eggshell membrane (ESM) by a green solvent-based electrospinning technique. Various analyses, including SEM, XRD, FT-IR, and TGA measurements, and tests for water solubility, wettability, water absorption and retention, antioxidant activity, and antibacterial properties were performed. SEM analysis showed average diameters from 257 to 325 nm. XRD results indicated decreased crystallinity after cross-linking. FT-IR measurements confirmed ester and acetal cross-link formation. TGA demonstrated enhanced thermal stability in cross-linked samples, especially PVA10%_lemon/esm10/honey20-H. Water solubility tests showed that heated samples were more stable. Water absorption rates exceeded 400%, with PVA10%_lemon/esm10/honey20-H having the highest retention rate. Wettability analysis showed significant changes in contact angles after heating. Antioxidant assays revealed that PVA10%_lemon had the highest DPPH activity (71.2%) among unheated samples, decreasing after cross-linking. Antibacterial tests showed significant activity only in PVA10%_lemon/esm10/honey20, against both *Escherichia coli* and *Bacillus subtilis* bacteria. Active ingredients can be added directly to this facial mask. This facial mask is gentler on the skin, and its ingredients have antiaging and anti-inflammatory properties. This mask can avoid the use of preservatives. This prepared facial mask has potential to be used in the organic skincare product industry and can also help the chemical industry toward sustainable and healthy practices.



1. INTRODUCTION

The skin is the largest organ in the human body, accounting for approximately 16% of its total weight.[1] The skin is composed of three layers: the subcutaneous tissue, the dermis, and the epidermis. Aging is caused by both endogenous and extrinsic determinants, which contribute to the progression of skin aging and the loss of physiological functions. Intrinsic factors include age and hormonal changes in skin tissue, while extrinsic factors include smoking, ultraviolet rays, and lifestyle choices.[2] In today's world, social media has expanded people's perspectives and significantly influenced beauty standards, leading to a growing awareness of facial care so the demand in the global face pack market has increased.[3] According to a study by Transparency Market Research, the global face pack market reached an estimated $713 million by 2031. This growth is attributed to the increasing incidence of skin diseases and a growing interest in maintaining beautiful skin.[3] According to NPD Group's Facial Skin Care Consumer Report, which surveyed female users, 46% of respondents reported purchasing products that are free of sulfates, phthalates, and gluten, an increase of 6 percentage points over the past two years. Additionally, more than half of the women surveyed are looking for skin care products made with organic ingredients.[4] Face packs include various types such as nonwoven masks, pulp masks, hydrogel masks, biocellulose masks, knit masks, and ampule sheet masks, as well as bubbling sheet masks.[5,6]

Traditional wet masks primarily function through the essence of liquid that they contain; however, their efficacy is often limited due to the presence of additives like thickening agents, film formers, and ultraviolet absorbents, which can compromise safety, skin health, and convenience.[7−9] These masks typically incorporate components such as vitamins, antioxidants, oils, emulsifiers, and silicones to control moisture loss and improve skin texture. Despite their benefits, the synthetic nature of these additives has driven interest toward

Received: October 14, 2024
Revised: January 15, 2025
Accepted: February 5, 2025
Published: March 26, 2025

"green tech" solutions that favor natural and sustainable materials. Additionally, these facial masks need to be packed in nonbiodegradable plastic barrier packaging. After their use, landfills and incineration are the main end-of-life options. A dry facial mask would certainly be a great solution to these issues. First, dry masks can avoid the use of antibacterial agents and preservatives. Second, dry masks can be sealed in biodegradable-paper-based packaging. Then, they have more hygienic and convenient operation. Most importantly, they can save a lot of water and raw materials and reduce their waste, but they are costly and prone to active ingredient inactivation.[10−12] Electrospinning technology has been successfully adopted in the cosmetics industry, and it is used for face pack materials because it produces nanofibers with a high surface area to volume ratio, enhancing the contact between the skin and the face pack and improving the penetration of active ingredients.[13,14] Recently, nanofiber mats have seen a hike in the market. Nanofibers can be used in different applications including high performance apparel, sound-heat absorbing materials, wound treatment, face masks, inorganic materials, oil-absorbing materials, and insulation materials.[15,16] The unique properties of nanofibers, such as a large surface area for their weight, give them excellent adsorption abilities. The supramolecular arrangement effect has a high strength due to the straight alignment of polymer chains. The dense structure leads to improved heat resistance.[17] The high surface to volume ratio of nanofibers can increase the contact area between the skin surface and the face pack, thereby increasing skin penetration.[18] Additionally, the nanofibers' fine structure can provide better adhesion to the skin and deliver ingredients more effectively.[19]

Poly(vinyl alcohol) (PVA) nanofibers are widely used in pharmaceuticals,[20] antibacterial treatments,[21] tissue engineering, and other applications due to their biodegradability, biocompatibility, nontoxicity, and low cost. As an excellent film-forming agent, PVA creates a protective and moisturizing barrier on the skin. The eggshell membrane, rich in bioactive compounds such as collagen, elastin, glucosamine, chondroitin sulfate, hyaluronic acid, keratin, and sulfur-rich proteins, enhances these benefits. It contains over 500 proteins, including various structural proteins,[22,23] which collectively improve skin hydration, elasticity, and reduce inflammation, promoting antiaging effects.[24,25] Additionally, it stimulates the production of type III collagen, further contributing to the skin's antiaging properties and overall health.[26−28] Honey, renowned for its historical use in skincare, adds significant advantages to this formulation. It primarily consists of sugars like fructose and glucose, along with proteins, amino acids, vitamins (ascorbic acid, biotin, nicotinic acid, pantothenic acid, pyridoxine, and thiamine), and enzymes (diastase, invertase, glucose oxidase, and catalase). Additionally, honey contains minerals such as potassium, magnesium, phosphorus, calcium, iron, and copper, as well as various plant-derived compounds.[29,30] These components make honey packed with antioxidants, enzymes, and nutrients that nourish and revitalize the skin. Its natural antibacterial properties help to cleanse and prevent acne, while its humectant nature retains moisture, keeping the skin hydrated and soft.[31,32] The phenolic acids, flavonoids, and proteins in honey exhibit antibacterial properties by exerting osmotic pressure on bacterial cells, thus inhibiting their growth.[33] Its anti-inflammatory properties soothe irritated skin and promote healing, making honey a valuable component in face packs. The long history of honey in beauty treatments attests to its efficacy in enhancing skin health and appearance.[34,35] For cross-linking the PVA/eggshell membrane-honey nanofiber, we use lemon juice. Lemon is rich in vitamin C and citric acid, which not only act as natural cross-linking agents but also provide additional skincare benefits.[36,37] Vitamin C helps in collagen synthesis, brightening the skin, and reducing dark spots, while citric acid acts as a gentle exfoliant, removing dead skin cells and promoting a smoother, more radiant complexion.[38] The incorporation of lemon juice enhances the overall efficacy of the skin pack, making it a comprehensive solution for moisturizing, antiaging, and skin brightening.[39,40]

Face packs contain additives, such as thickeners and preservatives, which have an adverse effect on the skin, and there is a growing demand for products made from organic products. To meet the demand, in this study, we fabricated a PVA nanofibrous facial mask that contains all-natural ingredients such as an eggshell membrane, lemon, and honey using an electrospinning technique. Furthermore, active ingredients can be added directly to this face mask. This allows face packs to be made without additives. Additionally, the high specific surface area of nanofibers can help with more skin penetration of the active ingredients. From these, the purpose of this research is to create a face pack that is gentler on the skin.

2. EXPERIMENTAL SECTION

2.1. Materials. Poly(vinyl alcohol) (PVA, Mw 85,000−124,000, 87−89% hydrolyzed) was purchased from Sigma-Aldrich (USA), the eggshell membrane was sourced from Farm-Foods (Japan), lemons were obtained from Sunkist (USA), and honey was acquired from Dabur India Limited (India).

2.2. Preparation of Nanofiber Membranes. Four different solutions were prepared for the fabrication of nanofiber membranes, as detailed in Table 1. The schematic illustration for the fabrication of nanofiber membranes is shown in Figure 1. Initially, a solution of 10 wt % PVA was dissolved in water and stirred at 80 °C for 2 h. This sample was designated as PVA10%. For the second solution, 10 wt % PVA was dissolved in lemon juice under the same conditions, and this sample was named PVA10%_lemon. In the third solution, 10 wt % PVA and 30 wt % eggshell membrane were dissolved

Table 1. Electrospinning Solution Scheme

samples	PVA (wt %)	lemon (solvent)	water (solvent)	ESM (wt %)	honey (wt %)
PVA10%	10		√		
PVA10%_lemon	10	√			
PVA10%_lemon/esm30	10	√		30	
PVA10%_lemon/esm10/honey20	10	√		10	20

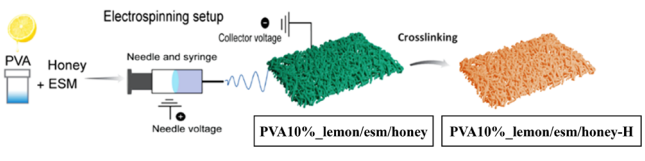


Figure 1. Schematic illustration for the preparation of the electrospun PVA_lemon/esm/honey nanofiber membrane.

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982

in lemon juice and stirred at 80 °C for 2 h, resulting in the sample PVA10%_lemon/esm30. Using the ESM within this range significantly improves the mechanical strength and bioactivity of the resulting nanofibers, making them suitable for various applications.[41] The fourth solution consisted of 10 wt % PVA, 10 wt % eggshell membrane, and 20 wt % honey dissolved in lemon juice, stirred at 80 °C for 2 h, and designated as PVA10%_lemon/esm10/honey20. A horizontal arrangement of high voltage power supply (Har-100*12, Matsusada Co., Tokyo, Japan), a rotating cylindrical collector, and a syringe pump (KDS-100, KD Scientific, USA) was set up for electrospinning. The spinning solution were filled in 20 mL plastic syringes fixed with 20-gauge needles. A high voltage of 15 kV was applied at the needle tip. The flow rate was kept at 0.7 mL/h. The distance between the needle tip and the collector was maintained at 15 cm. Nanofiber membranes were collected on butter paper wrapped around the collector. The electrospinning was carried out at an ambient temperature of 25 °C.

2.3. Cross-Linking. Poly(vinyl alcohol) (PVA) nanofibers can undergo cross-linking through thermal treatment without the need for additional chemical agents. This process involves heating the PVA nanofibers to a temperature that facilitates the formation of hydrogen bonds between hydroxyl groups on adjacent polymer chains, leading to a physically cross-linked network.[42] Three types of nanofibers, PVA10%_lemon, PVA10%_lemon/esm30, and PVA10%_lemon/esm10/honey20 were heated at 90 °C for 1 day to achieve cross-linking. The resulting cross-linked products were designated as PVA10%_lemon-H, PVA10%_lemon/esm30-H, and PVA10%_lemon/esm10/honey20-H, respectively.

2.4. Characterization. *2.4.1. Structural Characterization.* The surface morphology of the prepared nanofiber membranes was examined by using a scanning electron microscope (SEM; JSM-6010LA, JEOL Ltd., Japan) at an applied voltage of 10 kV, revealing detailed structural characteristics. To ensure conductivity, the nanofibers were coated with platinum by using a sputtering device (JFC-1600, JEOL, Japan). A total of 50 nanofibers were randomly selected from the SEM images, and the average fiber diameter was calculated from SEM images using ImageJ software. Chemical interactions within the samples were investigated using Fourier transform infrared spectroscopy (FT-IR; Shimadzu Corporation, Japan), with a range of 4000−600 cm^{-1} wavenumber, which analyzes the absorbed infrared light to identify molecular structures. The crystallinity of the samples was confirmed by using X-ray diffraction (XRD; Miniflex3000, Rigaku Co., Ltd., Japan), which provides diffraction patterns indicative of crystalline phases. The scanning speed was kept at 5°/min, and the 2θ range was between 5 and 60°. Additionally, thermogravimetric analysis (TGA; TGA8120, Rigaku Co., Ltd., Japan) was performed to assess the thermal stability and composition of the samples by measuring weight changes over a temperature range of 0 to 500 °C with a heating rate of 10 °C/min up to 600 °C.

2.4.2. Water Solubility, Absorption, and Retention. The water-related properties of the nanofiber samples were thoroughly evaluated through solubility, absorption, and retention tests. For the water solubility test, all nanofiber samples were cut into 2 cm × 2 cm pieces, placed in vials containing distilled water, and observed for dissolution. The water absorption capacity was tested by immersing 20 mg samples of the cross-linked nanofibers (PVA10%_lemon-H, PVA10%_lemon/esm30-H, and PVA10%_lemon/esm10/honey20-H) in phosphate-buffered saline (0.01 M, pH 7.2−7.4) for 40 or 120 s to absorb surface moisture, followed by weight measurement. The water absorption rate was calculated using the following formula:

$$\text{water absorption } (\%) = \frac{m - n}{m} \times 100 \tag{1}$$

where m is the initial weight and n is the final weight. The water retention test involved immersing the same types of 20 mg samples in phosphate-buffered saline for 40 s to absorb moisture and then measuring the weight at 15 min intervals over a 60 min period. The water retention rate was calculated using the following formula:

$$\text{water retention } (\%) = \frac{m - t}{m} \times 100 \tag{2}$$

where m is the initial weight and t is the weight after a specified time.

2.4.3. Surface Wettability Test. The surface wettability of the fabricated nanofiber samples was evaluated by measuring the contact angle. This test involved placing a droplet of liquid on the solid surface of the nanofibers and observing the droplet's behavior. The contact angle, which is the angle between the liquid droplet and the solid surface, was calculated using Young's equation:

$$\gamma S = \gamma L \cdot \cos\theta + \gamma SL \tag{3}$$

where γS is the surface tension of the solid, γL is the surface tension of the liquid, and γSL is the interfacial tension between the solid and the liquid.

$$\tan\theta_1 = \frac{h}{r} \rightarrow \theta = 2arc\tan\frac{h}{r} \tag{4}$$

with h representing the height and r the radius of the droplet. Each sample was measured five times to ensure accuracy, and the average contact angle was calculated. This analysis provided insights into the hydrophilicity and potential skin interaction properties of the nanofiber membranes.

2.4.4. Antioxidant and Antibacterial Properties. The antioxidant and antibacterial properties of the nanofiber samples were rigorously assessed. The antioxidant activity was measured using a 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical test. Samples were immersed in an 0.1 mM DPPH ethanol solution and incubated for 30 min in the dark at 37 °C. The absorbance at 517 nm was measured using UV−vis spectroscopy, and the antioxidant activity was calculated using the following formula[55]

$$\text{antioxidant activity } (\%) = \frac{Ac - As}{Ac} \times 100 \tag{5}$$

where As is the absorbance with the sample and Ac is the absorbance without the sample. For antibacterial testing, the nanofibers' properties were evaluated using *Escherichia coli* and *Bacillus subtilis*. After culturing the bacteria for 24 h, 30 μL of each bacterium was sprayed on agar medium. The samples, sterilized with ultraviolet light for 6 h, were cut to a diameter of 10 mm and placed on the agar medium and then incubated for 24 h. The presence or absence of antibacterial properties was determined based on the bacterial growth around the samples.

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982

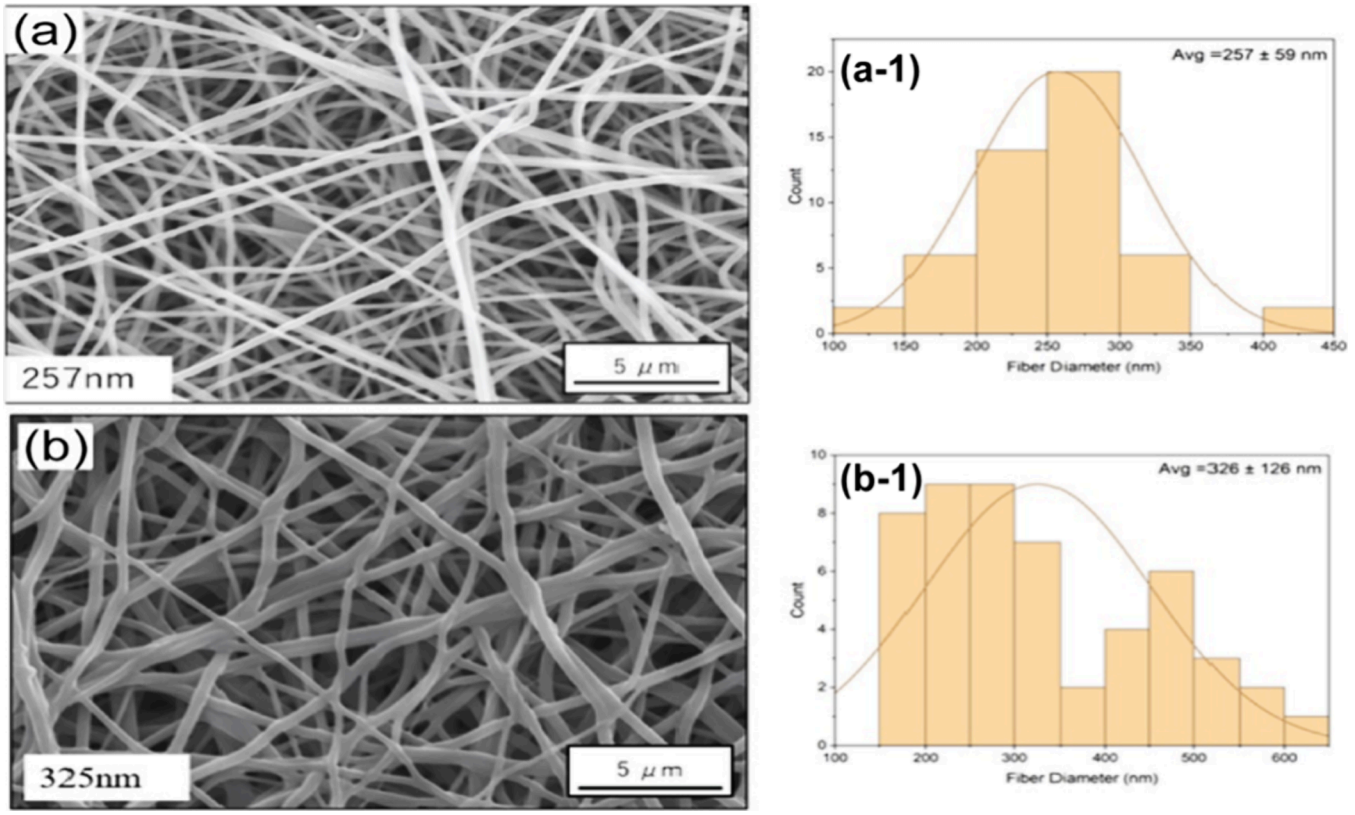


Figure 2. SEM images of (a) PVA10% and (b) PVA10%/esm30 in water and nanofiber diameter histograms of (a-1) PVA10% and (b-1) PVA10%/esm30.

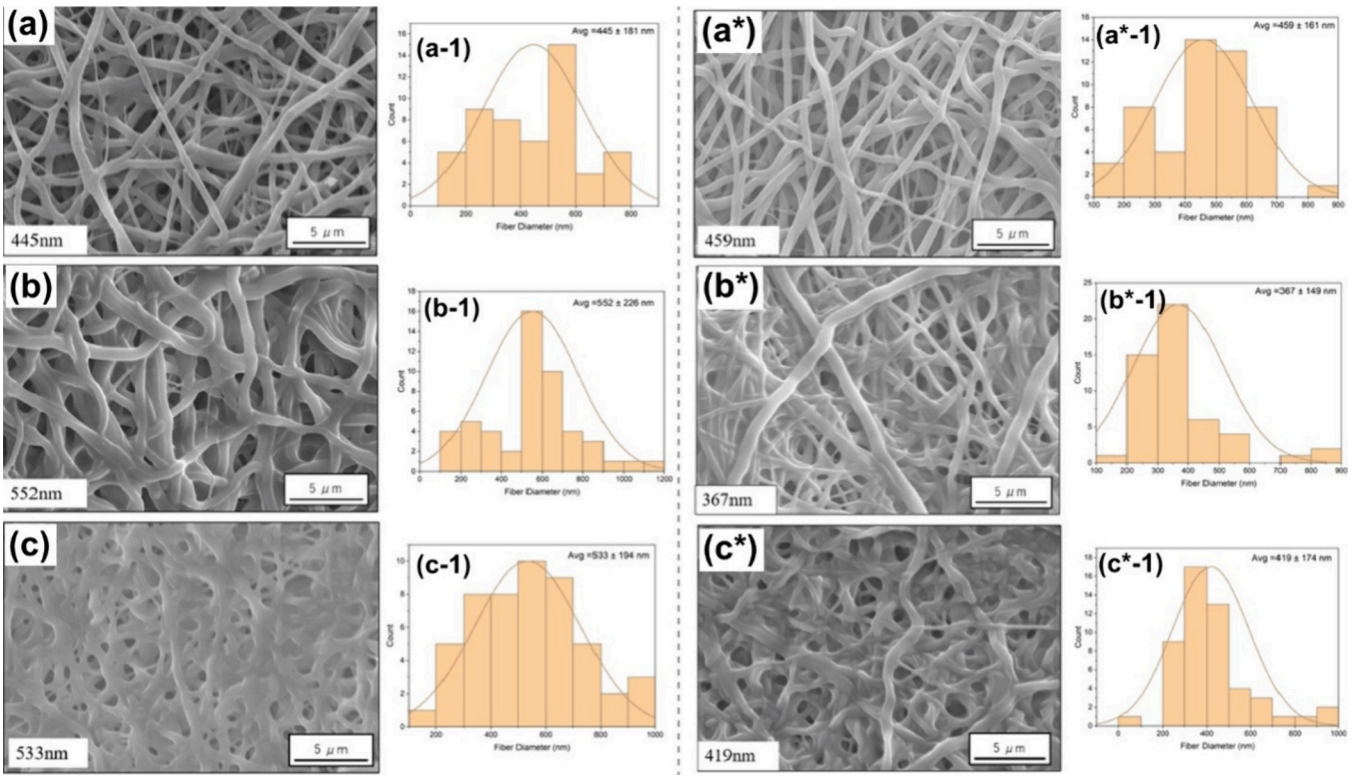


Figure 3. SEM images (a, b, and c) and its corresponding nanofiber diameter histograms (a-1, b-1, and c-1) of PVA10%_lemon, PVA10%_lemon/esm30, and PVA10%_lemon/esm10/honey20. SEM images (a*, b*, and c*) and its corresponding nanofiber diameter histograms (a*-1, b*-1, and c*-1) of PVA10%_lemon-H, PVA10%_lemon/esm30-H, and PVA10%_lemon/esm10/honey20-H.

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982

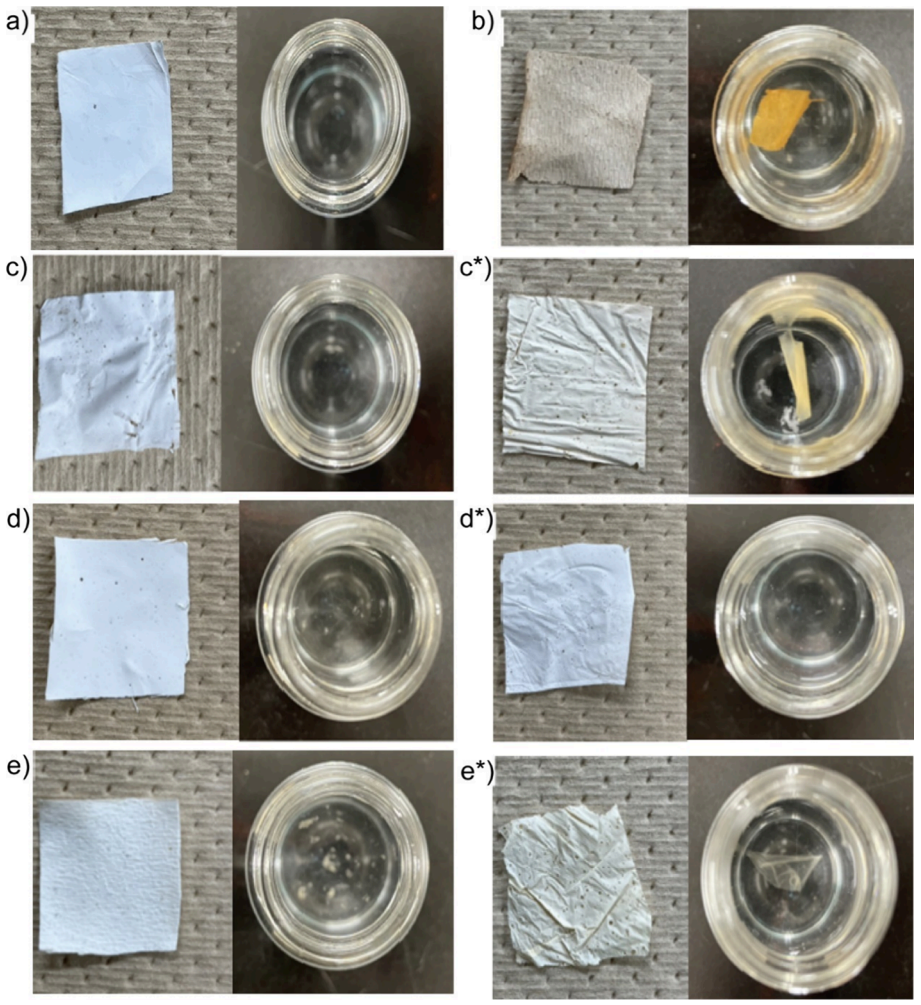


Figure 4. Water solubility test of (a) PVA10%, (b) PVA10%_esm30-H, (c) PVA10%_lemon, (c*) PVA10%_lemon-H, (d) PVA10%_lemon/esm30, (d*) PVA10%_lemon/esm30-H, (e) PVA10%_lemon/esm10/honey20, and (e*) PVA10%_lemon/esm10/honey20-H. The samples are 2 × 2 cm.

3. RESULTS AND DISCUSSION

3.1. Surface Morphology. The nanofiber membranes were successfully obtained. The SEM images and fiber diameter histograms of PVA10%_lemon and PVA10%_lemon/esm30 in water are depicted in Figure 2. The nanofiber membranes showed smooth, uniform, and beadless morphology. The average fiber diameter for the PVA10%_lemon sample is 257 nm, whereas it increases to 325 nm in the PVA10%_lemon/esm30 composite. This increase suggests that incorporating ESM into the PVA matrix influences fiber formation, potentially due to an increase in solution viscosity and molecular interactions between PVA and the ESM that results in increased fiber diameter.[43] Figure 3 further depicts the SEM images and diameter histograms of PVA10%_lemon, PVA10%_lemon/esm30, and PVA10%_lemon/esm10/honey20 before and after cross-linking. The nanofiber membranes showed thicker fibers with dense morphology especially in the case of honey-incorporated nanofiber membranes. The fiber diameters were 445, 552, and 533 nm, respectively, which shifted to 459, 367, and 419 nm after cross-linking. The fabricated nanofibers, with diameters below 550 nm, are considerably thinner than typical face pack materials, indicating enhanced skin permeability.[44] Fibers within this range are fine enough to adhere closely to the skin, ensuring a uniform contact surface and effective delivery of active ingredients. Fibers in this diameter range create an interconnected porous structure that balances breathability and occlusion, making the mask comfortable to wear while allowing moisture retention.[45] Notably, the composite fibers incorporating PVA, honey, and ESM exhibited larger diameters compared to pure PVA fibers, likely due to the increased viscosity of the spinning solution, which influences the stretching and thinning of the jet during electrospinning. Higher viscosity can reduce the extent of jet elongation, resulting in thicker fibers.[46] These interconnected nanofibrous membranes hold significant potential for applications such as wound dressings, facial masks, and adsorption systems.[47]

3.2. Water Solubility Analysis. The water resistance, thermal stability, and mechanical robustness of PVA nanofibers are essential for diverse applications, particularly in the biomedical field.[48] To evaluate the water stability, the composite nanofibers were subjected to solubility tests both before and after thermal cross-linking. The results of the water solubility test are depicted in Figure 4, which revealed that unheated samples dissolved completely in water, rendering the nanofibers invisible. Interestingly, the PVA10%_esm30 sample displayed inherent water resistance even without heating, attributed to the incorporation of the ESM. Conversely,

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982

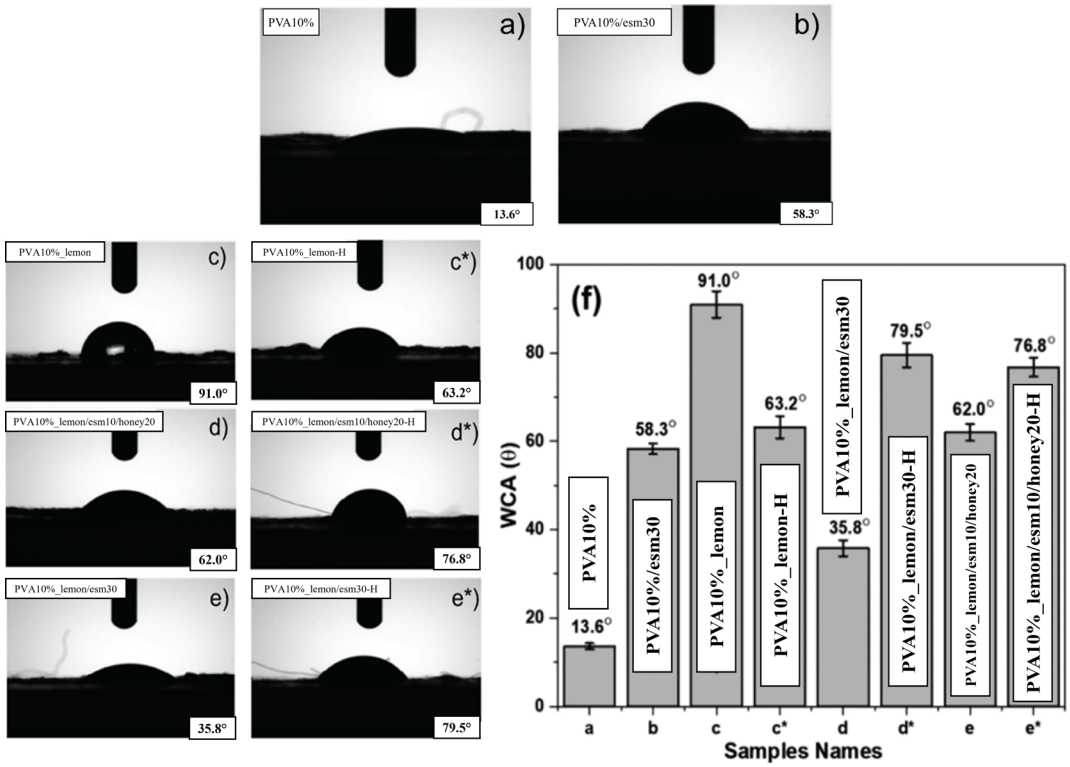


Figure 5. Water contact angle of the nanofiber membrane before (a, b, c, d, and e) and after (c*, d*, and e*) cross-linking samples and (f) WCA measurements in bar graph representation with an error bar.

thermally cross-linked samples such as PVA10%_lemon-H (c*) and PVA10%_lemon/esm10/honey20-H (e*) retained their structural integrity and remained visible in water, demonstrating the effectiveness of the cross-linking process. These cross-linked nanofibers exhibited remarkable water stability, lasting up to 1 day. Enhanced stability is likely due to the formation of robust intermolecular bonds during thermal cross-linking of PVA with citric acid and other natural additives, such as lemon and honey.[49] This cross-linked network strengthens the nanofiber structure, preventing dissolution by inhibiting the disintegration of PVA chains.[50]

3.3. Wettability Analysis. The wettability of all nanofiber membranes was investigated by measuring the water contact angle (WCA). Generally, a WCA value that is ≤90° indicates the hydrophilic nature of the nanofiber surface, and a WCA value ≥90° of the nanofiber surface is considered hydrophobic. Additionally, other factors are also responsible for the WCA of the surface such as the chemical composition of the nanofibers, roughness of the nanofiber surface, and surface free energy.[41] Wettability analysis was conducted to assess the hydrophilic or hydrophobic nature of the PVA nanofiber composites before and after thermal cross-linking, which is crucial for understanding their interaction with water. The water contact angle (WCA) measurements for the nanofiber samples, as shown in Figure 5, provide insight into their surface properties. For the unheated samples, the contact angles indicated varying degrees of hydrophilicity and hydrophobicity. Pure 10%PVA exhibited a highly hydrophilic nature, with a contact angle of 13.6°. The addition of the ESM increased the hydrophobicity to 58.3°. Incorporating lemon further shifted the contact angle to 91.0°, indicating hydrophobicity. Adding the ESM and lemon together resulted in a composite with a contact angle of 35.8°, balancing between the hydrophilic and hydrophobic properties. The addition of honey to this composite increased the contact angle to 62.0°. After thermal cross-linking, significant changes in contact angles were observed. The PVA10%_lemon-H sample showed a decrease in the contact angle by approximately 30.55%, indicating increased hydrophilicity. Lemon extract contains hydrophilic components, such as polyphenols. During the heating process, these compounds may migrate to the fiber surface, enhancing hydrophilicity and further reducing the WCA.[51] The PVA10%_lemon/esm30-H sample exhibited a substantial increase in contact angle by approximately 122.32%, suggesting a dramatic shift toward hydrophobicity. The PVA10%_lemonesm10/honey20-H sample also showed an increase in contact angle by approximately 23.23%, reflecting enhanced hydrophobic properties after cross-linking.[52] These results suggest that the hydrophilicity of PVA nanofiber composites is significantly influenced by the addition of the ESM, lemon, and honey, as well as by the thermal cross-linking process. Unheated samples are generally more hydrophilic, with pure PVA showing the highest water affinity. The addition of lemon and honey increases hydrophobicity, particularly after cross-linking, which reduces the availability of hydrophilic groups on the nanofiber surfaces. This shift toward increased hydrophobicity in heated samples indicates successful cross-linking and potential applications where moisture resistance and durability are desired.[53,54]

3.4. Water Absorption and Retention Analysis. To study the application of nanofiber facial masks, the water absorption and retention ability of these prepared nanofiber membranes were investigated for the evaluation of the moisturizing capacity.[55] A water absorption test was conducted on three cross-linked samples, PVA10%_lemon-H, PVA10%_lemon/esm30-H, and PVA10%_lemon/esm10/honey20-H,

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982

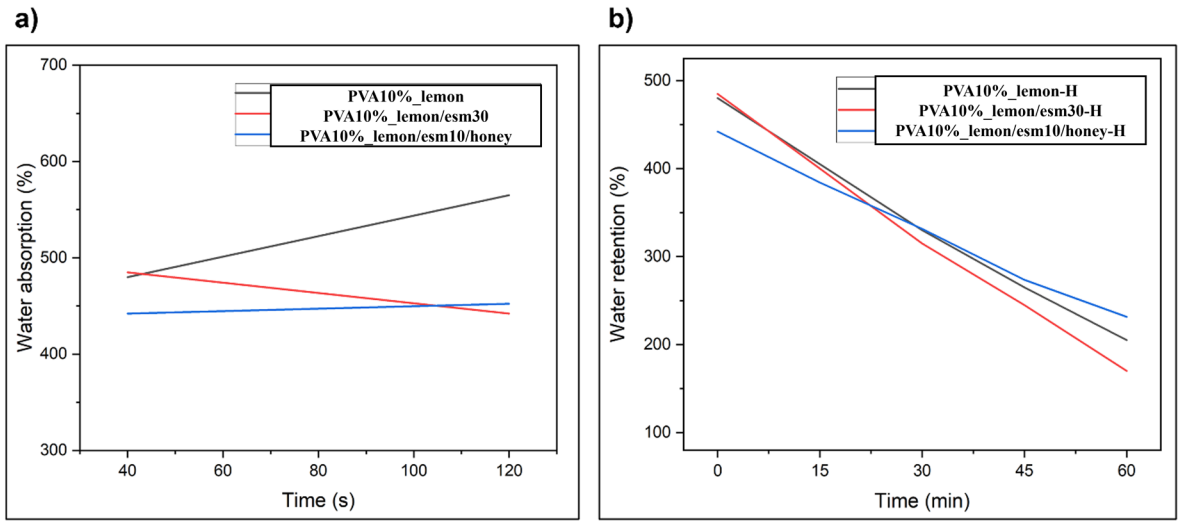


Figure 6. (a) Water absorption rates of cross-linked nanofiber samples PVA10%_lemon-H, PVA10%_lemon/esm30-H, and PVA10%_lemon/esm10/honey20-H over time and (b) water retention rates of the same cross-linked nanofiber samples.

as depicted in Figure 6a. Among these, PVA10%_lemon/esm30-H and PVA10%_lemon/esm10/honey20-H displayed distinct behaviors, with maximum water absorption achieved within 40 s. All samples exhibited water absorption rates exceeding 400%, which is ideal for facial mask applications. This high absorption capacity is primarily attributed to the nanofibers' large specific surface area. A water retention test, shown in Figure 6b, demonstrated that PVA10%_lemon/esm10/honey20-H had the lowest initial absorption rate but the highest retention rate after 60 min, indicating superior moisture retention capabilities. These variations in absorption and retention are attributed to differences in the composition and structure of the nanofibers.[56] Cross-linking modifies intermolecular forces, porosity, and surface characteristics, influencing water interaction. Furthermore, the addition of lemon and honey components enhances the hydrophilicity and water-trapping ability, thereby affecting the performance. These findings emphasize the potential for customizing nanofiber properties for advanced applications in materials science and biomedical engineering.[57,58]

3.5. Antioxidant Test. DPPH and hydroxyl (HO•) free radical scavenging assays are useful ways to characterize antioxidant activity. The scavenging free radical ability of nanofibers toward DPPH and hydroxyl radicals was assessed. The antioxidant activity results indicate that unheated samples consistently exhibit higher DPPH radical scavenging activity compared to their heated counterparts, as depicted in Figure 7. Pure PVA10% demonstrated no antioxidant properties; however, incorporating the ESM, lemon, and honey markedly improved the DPPH activity. Among the unheated samples, PVA10%_lemon exhibited the highest activity at 71.2%, followed by PVA10%_/esm10/honey20 at 56.4% and PVA10%_lemon/esm30 at 39.1%. In contrast, PVA10%/esm30 displayed the lowest activity, at 12.5%. The enhanced antioxidant activity in lemon- and honey-enriched samples is attributed to natural antioxidants, including vitamin C, polyphenols, flavonoids in lemon, and phenolic compounds in honey, which possess potent radical scavenging properties.[59] Upon thermal cross-linking, antioxidant activity declined in all samples, with PVA10%_lemon/esm30-H showing the highest DPPH activity at 42.1%, followed by PVA10%/esm30/honey20-H at 22.4% and PVA10%_lemon-H at 19.3%. The

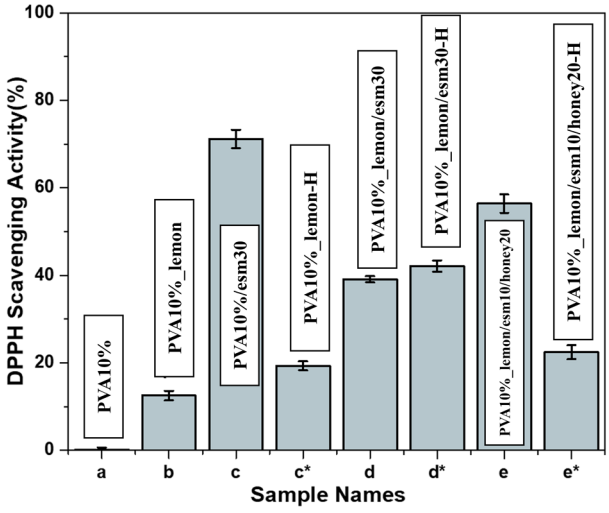


Figure 7. Antioxidant activity of PVA nanofiber composites as measured by the DPPH radical scavenging assay.

reduction is likely due to thermal degradation or structural alterations of the antioxidant components, impairing their efficiency. Additionally, the cross-linking process may limit the availability and mobility of these active compounds, reducing their radical scavenging potential. These findings underscore the significant enhancement of antioxidant properties achieved by incorporating natural additives such as lemon and honey. However, optimizing cross-linking conditions and using thermally stable antioxidants could be crucial to retaining superior antioxidant performance in the final nanofiber composites.[60,61]

3.6. Antibacterial Test. The antibacterial activity of the fabricated nanofibers was assessed against *B. subtilis* (a representative Gram-positive bacterium) and *E. coli* (a representative Gram-negative bacterium). Among the samples evaluated, only the honey-containing composite, PVA10%/esm10/honey20, demonstrated antibacterial efficacy. The inhibition zones, reflecting bacterial viability suppression, measured 1.1 mm for *E. coli* and 3.2 mm for *B. subtilis*, as illustrated in Figure 8. Th MIC of Gram-positive bacteria is often lower (5−20%), while the MIC of Gram-negative

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982

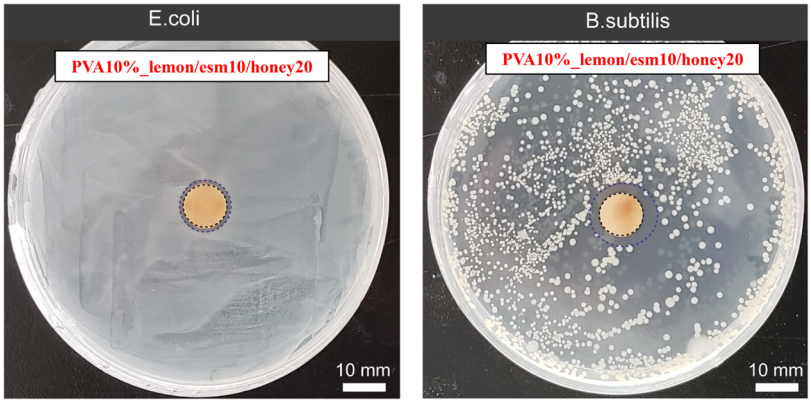


Figure 8. Zones of inhibition on agar plates demonstrating the antibacterial activity of the PVA10%_lemon/esm10/honey20 nanofiber composite.

Table 2. Comparison of Developed PVA Nanofiber Composites with Related Literature

property	developed nanofibers	related literature	reference
nanofiber diameter	300−500 nm	Electrospun nanofibers for skincare applications typically exhibit diameters in the range of 200−500 nm	45
thermal stability	enhanced in cross-linked samples, particularly in PVA10%_lemon/esm10/honey20-H	Cross-linking methods, such as heat treatment, have been shown to improve the thermal stability of electrospun nanofibers.	5 (SI)
water retention	>400%, highest in PVA10%_lemon/esm10/honey20-H	Nanofibers incorporating hydrophilic agents like hyaluronic acid or glycerin have demonstrated similar or higher water retention capacities.	57
antioxidant activity	71.2% DPPH activity (PVA10%_lemon)	Nanofibers containing natural antioxidants, such as grape seed extract, have shown DPPH radical scavenging activities ranging from 60 to 85%.	59
antibacterial activity	inhibition zones: 1.1 mm (*E. coli*), 3.2 mm (*B. subtilis*)	Electrospun nanofibers loaded with honey have exhibited significant antibacterial properties against both Gram-positive and Gram-negative bacteria.	65
wettability	contact angle decreased by 30.55% for PVA_lemon, increased by 122.32% (PVA_lemon/esm30)	Modifications in nanofiber composition, such as the incorporation of hydrophilic or hydrophobic substances, can significantly alter wettability and contact angles.	66

bacteria is typically higher (20−50%) due to their outer membrane providing additional resistance.[62] These findings highlight that incorporating honey into the PVA_lemon/esm nanofiber significantly enhances antibacterial properties, particularly against Gram-positive bacteria. The antibacterial effect of honey can be attributed to its natural antimicrobial agents, including hydrogen peroxide, phenolics, and flavonoids, which are known to disrupt bacterial growth and viability.[63] These results underscore the potential of honey-infused PVA nanofiber composites for applications requiring robust antibacterial functionality.[64]

4. CONCLUSIONS

This study successfully developed poly(vinyl alcohol) (PVA) nanofiber composites incorporating an eggshell membrane (ESM), lemon extract, and honey using an electrospinning technique (Table 2). SEM analysis revealed that the nanofiber diameters ranged from 257 nm for 10%PVA to 325 nm for PVA10%/esm30. Cross-linking improved the thermal stability, especially in the PVA10%_lemon/esm10/honey20-H composite. Water solubility tests showed enhanced water resistance in heated samples, with all samples absorbing over 400%, and the PVA10%_lemon/esm10/honey20-H sample exhibited the highest water retention, indicating moisturizing potential. Wettability tests showed a decrease in contact angles for PVA_lemon and increases for PVA10%_lemon/esm30 and PVA10%_lemon/esm10/honey20 after heating. The unheated PVA10%_lemon sample displayed the highest antioxidant activity, with 71.2% DPPH radical scavenging due to the antioxidant properties of lemon and honey. The PVA10%_lemon/esm10/honey20 composite also showed antibacterial activity, with inhibition zones of 1.1 mm for *E. coli* and 3.2 mm for *B. subtilis*. The PVA10%_lemon composite is ideal for antioxidant facial masks, while the PVA10%_lemon/esm10/honey20-H composite is recommended for hydrating and antibacterial applications. The PVA10%_lemon/esm30-H composite is suitable for applications that need improved water stability and wettability. In conclusion, the developed all-natural facial mask shows excellent properties for skin hydration, antioxidant protection, and antibacterial benefits, making it a sustainable, preservative-free option for improving skin health.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.4c09385.

Characterization results (XRD, FT-IR, and TGA measurements) of the nanofiber membrane (PDF)

■ AUTHOR INFORMATION

Corresponding Authors

Muhammad Farooq − *Nano Fusion Technology Research Group, Institute for Fiber Engineering and Science (IFES), Interdisciplinary Cluster for Cutting Edge Research (ICCER), Shinshu University, Ueda, Nagano 386-8567, Japan;* orcid.org/0009-0001-3427-3918; Email: farooq@shinshu-u.ac.jp

Ick Soo Kim − *Graduate School of Medicine, Science, and Technology and Nano Fusion Technology Research Group, Institute for Fiber Engineering and Science (IFES), Interdisciplinary Cluster for Cutting Edge Research (ICCER),*

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982

Shinshu University, Ueda, Nagano 386-8567, Japan; orcid.org/0000-0003-2126-0381; Phone: +81-268-21-5439; Email: kim@shinshu-u.ac.jp

Authors

Maira Khalid − *Graduate School of Medicine, Science, and Technology and Nano Fusion Technology Research Group, Institute for Fiber Engineering and Science (IFES), Interdisciplinary Cluster for Cutting Edge Research (ICCER), Shinshu University, Ueda, Nagano 386-8567, Japan*

Muhammad Adnan − *Graduate School of Medicine, Science, and Technology and Nano Fusion Technology Research Group, Institute for Fiber Engineering and Science (IFES), Interdisciplinary Cluster for Cutting Edge Research (ICCER), Shinshu University, Ueda, Nagano 386-8567, Japan*

Shoki Kobe − *Graduate School of Medicine, Science, and Technology and Nano Fusion Technology Research Group, Institute for Fiber Engineering and Science (IFES), Interdisciplinary Cluster for Cutting Edge Research (ICCER), Shinshu University, Ueda, Nagano 386-8567, Japan*

Gopiraman Mayakrishnan − *Nano Fusion Technology Research Group, Institute for Fiber Engineering and Science (IFES), Interdisciplinary Cluster for Cutting Edge Research (ICCER), Shinshu University, Ueda, Nagano 386-8567, Japan;* orcid.org/0000-0002-0137-8617

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.4c09385

Author Contributions

Maira Khalid: conceptualization, methodology, experimental, characterization, writing (original draft), investigation, visualization, formal analysis, data curation, writing (review and editing); Muhammad Farooq: conceptualization, methodology, writing (original draft), investigation, visualization, formal analysis, data curation, writing (review and editing), supervisor; Muhammad Adnan: visualization, formal analysis, data curation, experimental; Shoki Kobe: visualization, formal analysis; Gopiraman Mayakrishnan: visualization, formal analysis; Ick Soo Kim: validation, resource, supervisor.

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

This paper is based on results obtained from a project, JPNP 22021, commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

■ REFERENCES

(1) Rush, A.; Muir, M. Maintaining Skin Integrity in Bariatric Patients. *Br. J. Community Nurs.* **2012**, *17* (4), 154−159.

(2) Farage, M. A.; Miller, K. W.; Elsner, P.; Maibach, H. I. Intrinsic and Extrinsic Factors in Skin Ageing: A Review. *Int. J. Cosmet. Sci.* **2008**, *30* (2), 87−95.

(3) Devendra, N.; Amit, K.; Rajesh, K.; Abhishek, K. A. *Review on latest trend of herbal cosmetics.* Organ. by **2018**, 103.

(4) Surber, C.; Kottner, J. Skin Care Products: What Do They Promise, What Do They Deliver. *J. Tissue Viability* **2017**, *26* (1), 29−36.

(5) Siri, C.; Wattanutchariya, W. Development of Natural Facial Mask for Skincare from Local Materials. *IOP Conf. Ser.: Mater. Sci. Eng.* **2019**, *635*, No. 012003.

(6) Nilforoushzadeh, M. A.; Amirkhani, M. A.; Zarrintaj, P.; Salehi Moghaddam, A.; Mehrabi, T.; Alavi, S.; Mollapour Sisakht, M. Skin Care and Rejuvenation by Cosmeceutical Facial Mask. *J. Cosmet. Dermatol.* **2018**, *17* (5), 693−702.

(7) Morganti, P.; Morganti, G.; Chen, H. D.; Gagliardini, A. Beauty Mask: Market and Environment. *J. Clin. Cosmet. Dermatol.* **2019**, *3* (2), 2576−2826.

(8) Herman, A.; Herman, A. P.; Domagalska, B. W.; Młynarczyk, A. Essential Oils and Herbal Extracts as Antimicrobial Agents in Cosmetic Emulsion. *Indian J. Microbiol.* **2013**, *53*, 232−237.

(9) Velasco, M. V. R.; Vieira, R. P.; Fernandes, A. R.; Dario, M. F.; Pinto, C. A. S. de O.; Pedriali, C. A.; Kaneko, T. M.; Baby, A. R. Short-term Clinical of Peel-off Facial Mask Moisturizers. *Int. J. Cosmet. Sci.* **2014**, *36* (4), 355−360.

(10) Yang, Q.; Tian, Y.; Liu, Y.; Shi, W.; Guo, P.; Lei, P.; Yang, Q.; Zhu, C.; Zeng, R.; Zhang, C.; Qu, Y. A novel multi- functional skin-care dry mask based on Bletilla Striata polysaccharide electrospun nanofibers. *Int. J. Biol. Macromol.* **2024**, *282*, No. 136780.

(11) Meftahi, A.; Samyn, P.; Geravand, S. A.; Khajavi, R.; Alibkhshi, S.; Bechelany, M.; Barhoum, A. Nanocelluloses as skin biocompatible materials for skincare, cosmetics, and healthcare: Formulations, regulations, and emerging applications. *Carbohydr. Polym.* **2022**, *278*, No. 118956.

(12) Teno, J.; Pardo-Figuerez, M.; Hummel, N.; Bonin, V.; Fusco, A.; Ricci, C.; Donnarumma, G.; Coltelli, M. B.; Danti, S.; Lagaron, J. M. Preliminary studies on an innovative bioactive skin soluble beauty mask made by combining electrospinning and dry powder impregnation. *Cosmetics* **2020**, *7* (4), 96.

(13) Jin, S. G. Production and Application of Biomaterials Based on Polyvinyl Alcohol (PVA) as Wound Dressing. *Chem. - Asian J.* **2022**, *17* (21), No. e202200595.

(14) Kenawy, E.-R.; Abdel-Hay, F. I.; El-Newehy, M. H.; Wnek, G. E. Controlled Release of Ketoprofen from Electrospun Poly (Vinyl Alcohol) Nanofibers. *Mater. Sci. Eng., A* **2007**, *459* (1−2), 390−396.

(15) Fathi-Azarbayjani, A.; Qun, L.; Chan, Y. W.; Chan, S. Y. Novel Vitamin and Gold-Loaded Nanofiber Facial Mask for Topical Delivery. *Aaps Pharmscitech* **2010**, *11*, 1164−1170.

(16) Thompson, C. J.; Chase, G. G.; Yarin, A. L.; Reneker, D. H. Effects of Parameters on Nanofiber Diameter Determined from Electrospinning Model. *Polymer (Guildf).* **2007**, *48* (23), 6913−6922.

(17) Imran, M.; Yousaf, A. B.; Farooq, M.; Kausar, S.; Yasmeen, S.; Kasak, P. Visible Light-Induced H2 Production and Pollutant Degradation by Copper Oxide Nanosphere Embedded Zinc-Cadmium-Sulfide Composite. *Emergent Mater.* **2024**, *7*, 937.

(18) Mehta, P.; Picken, H.; White, C.; Howarth, K.; Langridge, K.; Nazari, K.; Taylor, P.; Qutachi, O.; Chang, M.; Ahmad, Z. Engineering Optimisation of Commercial Facemask Formulations Capable of Improving Skin Moisturisation. *Int. J. Cosmet. Sci.* **2019**, *41* (5), 462−471.

(19) Lin, J.-H.; Shiu, B.-C.; Hsu, P.-W.; Lou, C.-W.; Lin, J.-H. PVP/CS/Phyllanthus Emblica Nanofiber Membranes for Dry Facial Masks: Manufacturing Process and Evaluations. *Polymers (Basel).* **2022**, *14* (21), 4470.

(20) Taepaiboon, P.; Rungsardthong, U.; Supaphol, P. Drug-Loaded Electrospun Mats of Poly (Vinyl Alcohol) Fibres and Their Release Characteristics of Four Model Drugs. *Nanotechnology* **2006**, *17* (9), 2317.

(21) Keirouz, A.; Wang, Z.; Reddy, V. S.; Nagy, Z. K.; Vass, P.; Buzgo, M.; Ramakrishna, S.; Radacsi, N. The History of Electrospinning: Past, Present, and Future Developments. *Adv. Mater. Technol.* **2023**, *8* (11), 1−34.

(22) Kalman, D. S.; Hewlings, S. The Effect of Oral Hydrolyzed Eggshell Membrane on the Appearance of Hair, Skin, and Nails in Healthy Middle-aged Adults: A Randomized Double-blind Placebo-controlled Clinical Trial. *J. Cosmet. Dermatol.* **2020**, *19* (6), 1463−1472.

(23) Pasarin, D.; Enascuta, C. E.; Enache-Preoteasa, C.; Matei, C. B.; Ghizdareanu, A.-I. Combined Alkaline and Enzymatic Hydrolysis of Eggshell Membranes for Obtaining Ingredients for Food and Cosmetic Applications. *Chem. Proc.* **2023**, *13* (1), 20.

(24) Park, K.-M.; Yoo, J.-H.; Shin, Y.-J. Effects of Egg Shell Membrane Hydrolysates on Skin Whitening, Wound Healing, and UV-Protection. *Food Sci. Anim. Resour.* **2012**, *32* (3), 308−315.
(25) Yoo, J.; Park, K.; Yoo, Y.; Kim, J.; Yang, H.; Shin, Y. Effects of Egg Shell Membrane Hydrolysates on Anti-Inflammatory, Anti-Wrinkle, Anti-Microbial Activity and Moisture-Protection. *Korean J. food Sci. Anim. Resour.* **2014**, *34* (1), 26.
(26) Ohto-Fujita, E.; Shimizu, M.; Sano, S.; Kurimoto, M.; Yamazawa, K.; Atomi, T.; Sakurai, T.; Murakami, Y.; Takami, T.; Murakami, T.; Yoshimura, K.; Hasebe, Y.; Atomi, Y. Solubilized Eggshell Membrane Supplies a Type III Collagen-Rich Elastic Dermal Papilla. *Cell Tissue Res.* **2019**, *376*, 123−135.
(27) Picard, J.; Paul-Gardais, A.; Vedel, M. Sulfated Glycoproteins from Egg Shell Membranes and Hen Oviduct. Isolation and Characterization of Sulfated Glycopeptides (Author's Transl). *Biochim. Biophys. Acta* **1973**, *320* (2), 427−441.
(28) Baker, J. R.; Balch, D. A. A Study of the Organic Material of Hen's-Egg Shell. *Biochem. J.* **1962**, *82* (2), 352.
(29) Wong, M.; Hendrix, M. J. C.; von der Mark, K.; Little, C.; Stern, R. Collagen in the Egg Shell Membranes of the Hen. *Dev. Biol.* **1984**, *104* (1), 28−36.
(30) Burlando, B.; Cornara, L. Honey in Dermatology and Skin Care: A Review. *J. Cosmet. Dermatol.* **2013**, *12* (4), 306−313.
(31) Beretta, G.; Artali, R.; Caneva, E.; Orlandini, S.; Centini, M.; Facino, R. M. Quinoline Alkaloids in Honey: Further Analytical (HPLC-DAD-ESI-MS, Multidimensional Diffusion-Ordered NMR Spectroscopy), Theoretical and Chemometric Studies. *J. Pharm. Biomed. Anal.* **2009**, *50* (3), 432−439.
(32) Thiele, J. Benefits of Honey: Potency against Micro-Organisms and Its Use in Skin Care. *J. Apither.* **2022**, *9* (5).
(33) Almasaudi, S. The Antibacterial Activities of Honey. *Saudi J. Biol. Sci.* **2021**, *28* (4), 2188−2196.
(34) Rafique, S.; Hassan, S. M.; Mughal, S. S.; Hassan, S. K.; Shabbir, N.; Perveiz, S.; Mushtaq, M.; Farman, M. Biological Attributes of Lemon: A Review. *J. Addict. Med. Ther. Sci.* **2020**, *6* (1), 30−34.
(35) Ryan, E. M.; Duryee, M. J.; Hollins, A.; Dover, S. K.; Pirruccello, S.; Sayles, H.; Real, K. D.; Hunter, C. D.; Thiele, G. M.; Mikuls, T. R. Antioxidant Properties of Citric Acid Interfere with the Uricase-Based Measurement of Circulating Uric Acid. *J. Pharm. Biomed. Anal.* **2019**, *164*, 460−466.
(36) Dhanavade, M. J.; Jalkute, C. B.; Ghosh, J. S.; Sonawane, K. D. Study Antimicrobial Activity of Lemon (Citrus Lemon L.) Peel Extract. *Br. J. Pharmacol. Toxicol.* **2011**, *2* (3), 119−122.
(37) Bekkouch, O.; Zengin, G.; Harnafi, M.; Touiss, I.; Khoulati, A.; Saalaoui, E.; Harnafi, H.; Abdellattif, M. H.; Amrani, S. Anti-Inflammatory Study and Phytochemical Characterization of Zingiber Officinale Roscoe and Citrus Limon L. Juices and Their Formulation. *ACS omega* **2023**, *8* (30), 26715−26724.
(38) Gadhave, R. V.; Mahanwar, P. A.; Gadekar, P. T. Effect of Glutaraldehyde on Thermal and Mechanical Properties of Starch and Polyvinyl Alcohol Blends. *Des. Monomers Polym.* **2019**, *22* (1), 164−170.
(39) Aliasgharlou, N.; Sana, F. A.; Khoshbakht, S.; Zolfaghari, P.; Charkhian, H. Fabrication and Characterization of Boric Acid-Crosslinked Ethyl Cellulose and Polyvinyl Alcohol Films as Potential Drug Release Systems for Topical Drug Delivery. *Turk. J. Chem.* **2020**, *44* (6), 1723−1732.
(40) Jiang, K.; Wang, X. Preparation and Application of Maleic Acid Crosslinked Polyvinyl Alcohol/Mica Coating for Barrier Paper. *Prog. Org. Coatings* **2022**, *170*, No. 106937.
(41) Nataraj, D.; Reddy, R.; Reddy, N. Crosslinking Electrospun Poly (Vinyl) Alcohol Fibers with Citric Acid to Impart Aqueous Stability for Medical Applications. *Eur. Polym. J.* **2020**, *124*, No. 109484.
(42) Ghorbanzadeh Sheish, S.; Emadi, R.; Ahmadian, M.; Sadeghzade, S.; Tavangarian, F. Fabrication and characterization of polyvinylpyrrolidone-eggshell membrane-reduced graphene oxide nanofibers for tissue engineering applications. *Polymers* **2021**, *13* (6), 913.
(43) Tsioptsias, C.; Fardis, D.; Ntampou, X.; Tsivintzelis, I.; Panayiotou, C. Thermal behavior of poly (vinyl alcohol) in the form of physically crosslinked film. *Polymers* **2023**, *15* (8), 1843.
(44) Gilaberte, Y.; Prieto-Torres, L.; Pastushenko, I.; Juarranz, Á. Chapter 1 - Anatomy and Function of the Skin. In *Nanoscience in Dermatology*; Elsevier: 2016; pp 1−14.
(45) Rathna, G. V. N.; Jog, J. P.; Gaikwad, A. B. Development of non-woven nanofibers of egg albumen-poly (vinyl alcohol) blends: influence of solution properties on morphology of nanofibers. *Polym. J.* **2011**, *43* (7), 654−661.
(46) Wang, Y.; Wang, N.; Liu, W.; Cheng, Z.; Li, W.; Zhao, Y.; Zhu, H. Preparation and evaluation of Gastrodia elata polysaccharide loaded electrospinning nanofiber facial mask. *J. Appl. Polym. Sci.* **2024**, *141* (45), No. e56199.
(47) Ahmadi Bonakdar, M.; Rodrigue, D. Electrospinning: Processes, structures, and materials. *Macromol.* **2024**, *4* (1), 58−103.
(48) Zand, M.; Sepahvand, S.; khoshkhat, P.; Chamani, M.; Jonoobi, M.; Ashori, A. Preparation and characterization of poly (vinyl pyrrolidone)/cellulose nanofiber/Aloe Vera composites as a biocompatible hydrating facial mask. *Int. J. Biol. Macromol.* **2024**, *277*, No. 133846.
(49) Teixeira, M. A.; Amorim, M. T. P.; Felgueiras, H. P. Poly (vinyl alcohol)-based nanofibrous electrospun scaffolds for tissue engineering applications. *Polymers* **2020**, *12* (1), 7.
(50) Abou Zekry, S. S.; Abdellatif, A.; Azzazy, H. M. E. Fabrication of Pomegranate/Honey Nanofibers for Use as Antibacterial Wound Dressings. *Wound Med.* **2020**, *28*, No. 100181.
(51) Haider, M. K.; Ullah, A.; Sarwar, M. N.; Yamaguchi, T.; Wang, Q.; Ullah, S.; Park, S.; Kim, I. S. Fabricating antibacterial and antioxidant electrospun hydrophilic polyacrylonitrile nanofibers loaded with agnps by lignin-induced in-situ method. *Polymers* **2021**, *13*, 748.
(52) Beikzadeh, S.; Akbarinejad, A.; Swift, S.; Perera, J.; Kilmartin, P. A.; Travas-Sejdic, J. Cellulose acetate electrospun nanofibers encapsulating Lemon Myrtle essential oil as active agent with potent and sustainable antimicrobial activity. *React. Funct. Polym.* **2020**, *157*, No. 104769.
(53) Rao, R.; Jindal, A.; Bhogal, B.; Pai, S. B. Direct Immunofluorescence Microscopy of Skin Biopsy Samples Preserved in Honey. *J. Am. Acad. Dermatol.* **2017**, *76* (4), 761−763.
(54) Gope, A.; Mukhopadhyay, A.; Mukhopadhyay, O.; Chatterjee, J. Regenerative Repair of Full Thickness Skin Wound Assisted by Dual Crosslinking Percolative Gel Casting Maneuvered Alginate Hydrogel Embedded with Honey Ghee Blend Resembles Standard Cutaneous Properties. *J. Tissue Viability* **2022**, *31* (4), 657−672.
(55) Kharaghani, D.; Khan, M. Q.; Tamada, Y.; Ogasawara, H.; Inoue, Y.; Saito, Y.; Hashmi, M.; Kim, I. S. Fabrication of Electrospun Antibacterial PVA/Cs Nanofibers Loaded with CuNPs and AgNPs by an in-Situ Method. *Polym. Test.* **2018**, *72*, 315−321.
(56) Koosha, M.; Aalipour, H.; Sarraf Shirazi, M. J.; Jebali, A.; Chi, H.; Hamedi, S.; Wang, N.; Li, T.; Moravvej, H. Physically crosslinked chitosan/PVA hydrogels containing honey and allantoin with long-term biocompatibility for skin wound repair: An in vitro and in vivo study. *J. Funct. Biomater.* **2021**, *12* (4), 61.
(57) Parın, F. N.; Aydemir, Ç. İ.; Taner, G.; Yıldırım, K. Co-Electrospun-Electrosprayed PVA/Folic Acid Nanofibers for Transdermal Drug Delivery: Preparation, Characterization, and in Vitro Cytocompatibility. *J. Ind. Text.* **2022**, *51* (1_suppl), 1323S−1347S.
(58) Parvinzadeh Gashti, M.; Dehdast, S. A.; Berenjian, A.; Shabani, M.; Zarinabadi, E.; Chiari Fard, G. PDDA/Honey Antibacterial Nanofiber Composites for Diabetic Wound-Healing: Preparation, Characterization, and In Vivo Studies. *Gels* **2023**, *9* (3), 173.
(59) Becerril-Sánchez, A. L.; Quintero-Salazar, B.; Dublán-García, O.; Escalona-Buendía, H. B. Phenolic compounds in honey and their relationship with antioxidant activity, botanical origin, and color. *Antioxidants* **2021**, *10* (11), 1700.

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982

(60) Yang, M.; Zhao, T.; Xia, W.; Wei, K.; Li, R.; Jiang, W.; Zhou, C.; Ben, H.; Zhang, J.; Ramakrishna, S.; Long, Y. Z. In-situ electrospinning with precise deposition of antioxidant nanofiber facial mask loaded with Enteromorpha prolifera polysaccharides. *Int. J. Biol. Macromol.* **2024**, *257*, No. 128698.
(61) Goodge, K. E.; Alwala, W. A.; Frey, M. W. Nanoweb for Nanobugs: Nanofiber Filter Media for Face Masks. *ACS Appl. Nano Mater.* **2024**, *7* (6), 6120−6129.
(62) McLoone, P.; Warnock, M.; Fyfe, L. Honey: A Realistic Antimicrobial for Disorders of the Skin. *J. Microbiol. Immunol. Infect.* **2016**, *49* (2), 161−167.
(63) Albaridi, N. A. Antibacterial potency of honey. *Int. J. Microbiol.* **2019**, *2019* (1), No. 2464507.
(64) Ullah, A.; Ullah, S.; Khan, M. Q.; Hashmi, M.; Nam, P. D.; Kato, Y.; Tamada, Y.; Kim, I. S. Manuka Honey Incorporated Cellulose Acetate Nanofibrous Mats: Fabrication and in Vitro Evaluation as a Potential Wound Dressing. *Int. J. Biol. Macromol.* **2020**, *155*, 479−489.
(65) Jeong, S.; Oh, S. G. Antiacne effects of PVA/ZnO composite nanofibers crosslinked by citric acid for facial sheet masks. *Int. J. Polym. Sci.* **2022**, *2022* (1), No. 4694921.
(66) Tahir, R.; Albargi, H. B.; Ahmad, A.; Qadir, M. B.; Khaliq, Z.; Nazir, A.; Khalid, T.; Batool, M.; Arshad, S. N.; Jalalah, M.; Alsareii, S. A.; Harraz, F. A. Development of Sustainable Hydrophilic Azadirachta indica Loaded PVA Nanomembranes for Cosmetic Facemask Applications. *Membranes* **2023**, *13* (2), 156.

https://doi.org/10.1021/acsomega.4c09385
ACS Omega 2025, 10, 12972−12982