



A STUDY ABOUT SAFFLOWER OIL LOADED EMULSION ELECTROSPUN NANOFIBERS

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ABSTRACT: *In this study, it was aimed to achieve production and characterization of safflower oil-loaded core sheath nanofibers via emulsion electrospinning. For this aim, firstly, polyvinyl(alcohol) (PVA) based emulsion polymer solutions were prepared with different safflower oil concentrations. Then, polymer solution properties were determined in terms of the understanding effect of safflower oil concentrations on emulsion electrospun nanofiber morphology. Nanofiber production was carried out under the optimum process parameters for all samples. Lastly, safflower-loaded nanofibrous surfaces were characterized morphologically and chemically. Gas chromatography-mass spectrometry (GC-MS) was performed for determination of oil acids and phenolic compounds in safflower oil. It is thought that the nanofibrous surfaces designed and produced have a high potential for use as face masks for skin care.*

Keywords: *Safflower oil, nanofibers, emulsion electrospinning, skin care, face mask.*

STUDIJA O ELEKTROSPINSKIM NANOVLAKNIMA U EMULZIJI PUNJENIM ULJEM ŠAFRANA

APSTRAKT: *Cilj ovog istraživanja bio je da se postigne proizvodnja i karakterizacija nanovlakna omotača jezgra napunjenih šafranovim uljem putem emulzionog elektropredenja. Za ovaj cilj, prvo, pripremljeni su rastvori emulzionih polimera na bazi polivinila(alkohola) (PVA) sa različitim koncentracijama ulja šafranike. Zatim su određena svojstva rastvora polimera u smislu razumevanja uticaja koncentracija ulja šafranike na morfologiju emulzionih elektropsunovih nanovlakna. Proizvodnja nanovlakna je obavljena po optimalnim procesnim parametrima za sve uzorke. Na kraju, morfološki i hemijski su okarakterisane nanofibrozne površine napunjene šafranom. Za određivanje uljnih kiselina i fenolnih jedinjenja u ulju šafranike urađena je gasna hromatografija-masena spektrometrija (GC-MS). Smatra se da nanofibrozne površine dizajnirane i proizvedene imaju veliki potencijal za upotrebu kao maske za lice za negu kože.*



Ključne reči: ulje šafranike, nanovlakna, elektrospinoвање emulzije, nega kože, maska za lice.

1. INTRODUCTION

Safflower oil is widely used in the food, cosmetics, pharmaceutical, and food industries. Safflower oil offers the additional benefit of being more cost-effective to produce, making it a viable alternative for individuals who are unable to purchase more expensive options such as olive oil and other valuable oils [1]. The high content of linoleic acid and α -tocopherol in safflower oil enhances the quality and appearance of skin. The hair and skin benefits of safflower oil are attributed to its vitamin E content and its lightweight structure, which promotes easy absorption into the scalp [2].

Emulsion electrospinning is an alternative to coaxial for producing core sheath nanofibers. There is no need to modify the classical electrospinning setup to produce nanofibers in the core-shell structure by emulsion electrospinning. One of the most important points is to create stable emulsions. If water-in-oil or oil-in-water emulsions are not formed correctly, the core shell structure cannot be achieved. To make stable emulsions with multiphase systems, the HLB value of the surfactant and the ratio of the inner phase to the outer phase are very important [3]. The advantage of core-shell nanofibers over nanofibers produced by other methods such as blend and suspension is the long-term release of the additives in the form of controlled release [4].

Upon further examination of the literature, it is observed that the studies in which safflower oil and nanofibers are combined are quite limited. When the superior properties of safflower oil and nanofibers are combined, it is thought that this material will have potential for use in different application areas.

2. MATERIALS AND METHODS

2.1. Materials

In this study, polyvinyl alcohol ($M_n = 88,000$, 88% hydrolyzed) was used as the polymer, distilled water was used as a solvent, safflower oil (*Carthamus tinctorius* L.) was used as a natural agent, and surfactant (Cremophor RH40) was used as an emulsifier.

2.1. Methods

The first step in the study was the preparation of stable emulsions. For this purpose, all PVA polymer solutions were prepared at an equal concentration of 10 wt %. Safflower oil was added to the PVA polymer solutions at different concentrations, such as 2 wt %, 4 wt %, 6 wt %, and 8 wt %. Finally, equal amounts (2 wt %) of surfactant were added to each solution to form an emulsion and stirred at room temperature. Then, solution properties such as viscosity, conductivity (SelectaCD2005), and surface tension with the Wilhemly Plate method (Biolin Scientific Sigma 702) were measured.

Emulsion electrospinning was carried out under optimum process parameters such as voltage (22.4 Kv), feed rate (0.4 mL/h), and distance between electrodes (22 cm). Then, safflower loaded PVA nanofibers were characterized via SEM images as morphological

and structural. FT-IR analysis was performed to verify the safflower oil in nanofibrous structure. GC-MS was used to determine the presence of oil acids and phenolic compounds in safflower oil.

3. RESULTS AND DISCUSSIONS

3.1. Solution Properties Results

Table 1: GC-MS analyze result of safflower oil

Component Name	Area (%)
Tetradecanoic acid (Myristic acid)	0.05
9-Hexadecenoic acid (Palmitoleic acid)	0.03
Hexadecanoic acid (Palmitic acid)	5.67
9,10-Methylene hexadecanoic acid	0.02
Heptadecanoic acid (Margaric acid)	0.01
9,12-Octadecadienoic acid (Linoleic acid)	46.67
9-Octadecenoic acid (Oleic acid)	44.72
Octadecanoic acid (Stearic acid)	2.17
11-Eicosenoic acid	0.09
Eicosenoic acid (Arachidic acid)	0.14
13-Docosenoic acid (Erucic acid)	0.01
Docosanoic acid (Behenic acid)	0.05
Other components	0.36

In all, 13 components were identified as fatty acids in the safflower oil structure. Main constituents were determined linoleic acid (46.67%), oleic acid (44.72%), palmitic acid (5.67%) and stearic acid (2.17%), respectively. Polymer solution properties were measured to investigate the effect of safflower oil concentration on solution properties such as viscosity, conductivity and surface tension (Figure 1).

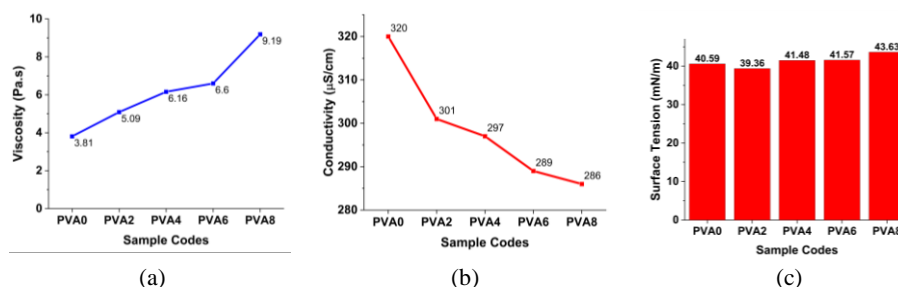
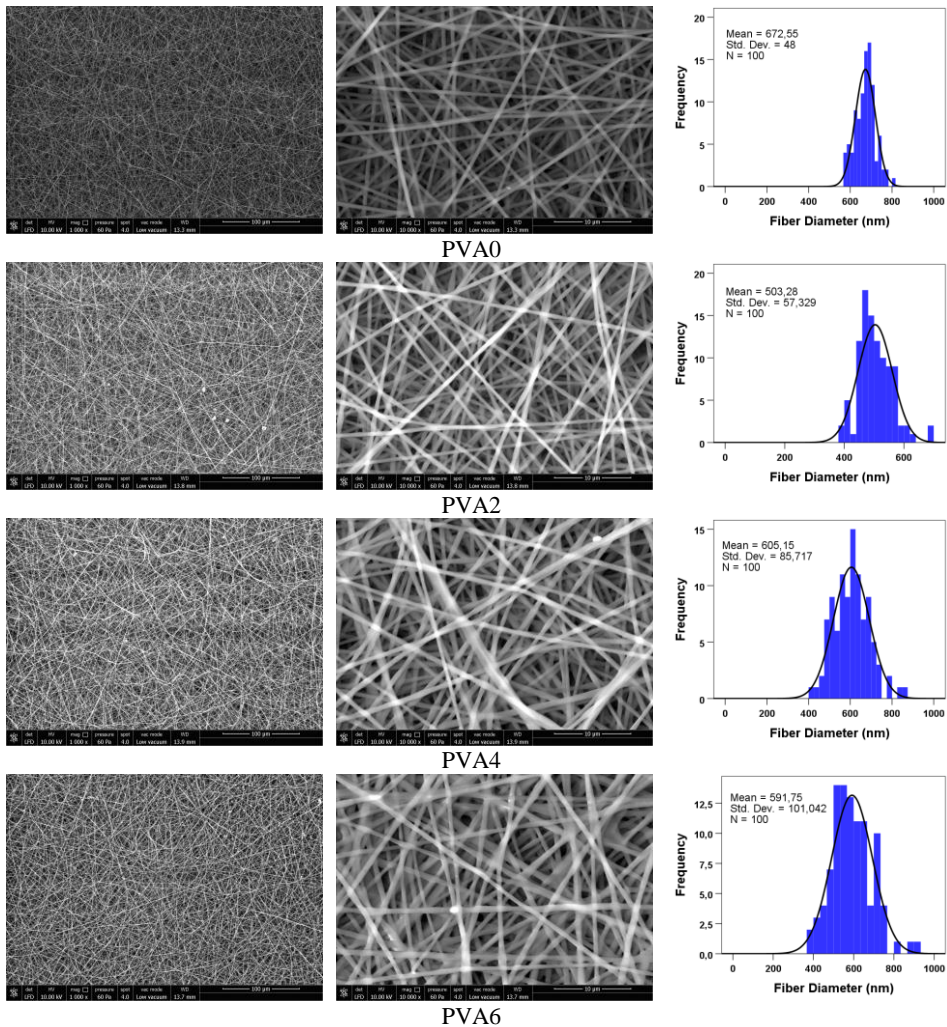


Figure 1: Solutions properties of PVA based polymer solutions with different concentrations of safflower oil a) Viscosity b) Conductivity c) Surface tension

When the graphs are analyzed, it is seen that as safflower oil concentration increases, viscosity increases, conductivity decreases and surface tension does not change.

3.2. Fiber Morphology Results

SEM images were taken to analyze the fiber morphology, fiber diameters and nanoweb quality. These SEM images are given in Figure 2 with fiber diameter histograms.



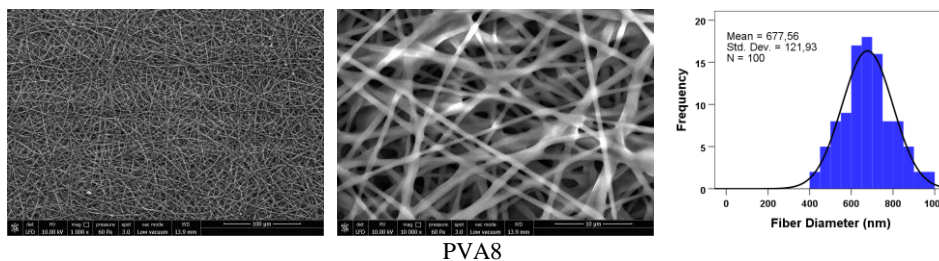


Figure 2: SEM images (1.000x-10.000x) and histograms of PVA nanofibers produced with various concentrations of safflower oil

As seen from SEM images and histograms, the nanofibers are fine, uniform, very smooth, bead-free, homogeneous, and high nanoweb quality. However, as the safflower oil concentration increased, reticulated structures were observed in the nanoweb structure. The normal distribution curve of all histograms has a single peak. This indicates that the nanofibers have a highly homogeneous fiber diameter distribution. Fiber diameter and fiber diameter uniformity coefficient with safflower oil concentration graph are given in Figure 3.

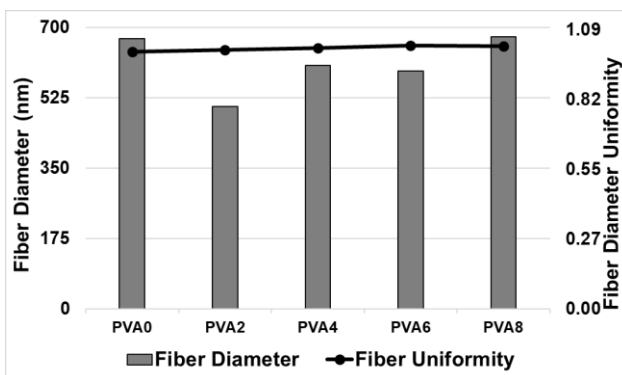


Figure 3: Average fiber diameter and diameter uniformity results of PVA nanofibers with various concentrations of safflower oil

According to Figure 3, average fiber diameter varies between 503 and 677 nm. Fiber diameter uniformity coefficients were close to 1 and were not affected by safflower oil concentration.

Chemical characterization was performed using Fourier transform infrared spectroscopy (FT-IR). FT-IR spectra of PVA, SA, and PCL10-1 nanofibers are given in Figure 3.

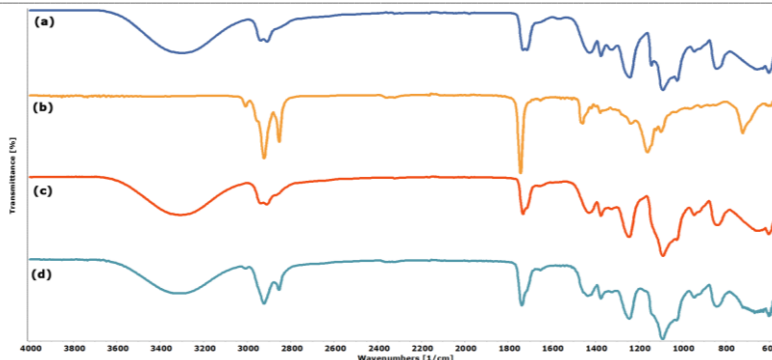


Figure 3: FT-IR spectra of: a) PVA; b) Safflower oil; c) PVA0; d) PVA8

In PVA spectra, the strong hydrogen bonded band occurred at approximately 3400-3200 cm^{-1} wavelength in the spectra of PVA polymer. Because of the high hydrophilic forces, intramolecular and intermolecular hydrogen bonding is expected to occur among PVA chains. Another important absorption peak was confirmed at 1141 cm^{-1} . The crystallinity of PVA is primarily responsible for this vibrational band, which is related to the carboxyl stretching band (C-O) [5]. These peaks appeared in the spectra of the PVA0 and PVA8 coded nanofibers. There are absorption bands in the general safflower oil spectrum at 1815–659, 3146–2816, 3391–3382, 3631–3530 cm^{-1} . Peaks around the 3600–3500 cm^{-1} region could be attributed to –OH stretching while the 3300–2800 cm^{-1} region is associated with –CH stretching. The C-O stretch takes place at 1800–1700 cm^{-1} and 1200–650 cm^{-1} is the finger-print region [6]. These characteristic peaks were not observed in the spectrum of the sample coded PVA0 without safflower oil, while the peaks appeared in the sample coded PVA8 containing 8 wt % safflower oil. This result chemically verified the presence of safflower oil in the structure of safflower oil loaded nanofibers.

4. CONCLUSION

In conclusion, the production and characterization of PVA-based nanofibers containing safflower oil were carried out by the emulsion electrospinning method. The findings of the study revealed the importance of the phases forming the emulsion in the emulsion electrospinning method. In other words, there was a strong relationship between oil concentration, solution properties, fiber spinning performance and fiber morphology.

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