Evaluation of aloe vera extract loaded polyvinyl alcohol nanofiber webs obtained via needleless electrospinning

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Abstract. In this study aloe vera liquid extract was added to PVA to obtain nanofiber mats with integrated bioactive compounds. Nanofibers are effective in drug release process and the most cost and time effective way to produce them is through roller electrospinning method. Several concentrations of PVA and aloe vera extract have been prepared, as well as pure 10 wt% polyvinyl alcohol solution to compare their properties. Viscosity and electroconductivity of solutions were measured, atomic force microscopy was used for evaluation of nanofiber mats morphology and diameter measurements, samples were analysed and compared by Fourier transform infrared spectroscopy and mechanical properties of the nanofiber mats were tested.

1. Introduction

Aloe vera is widely known for its health benefits. Since ancient times people used it topically to treat skin problems and to heal digestive disorders. Nowadays there are many consumer products available in different forms - gels, creams, drinks etc. [1]. Although, there are concerns regarding possible toxicity of orally administrated aloe vera [2], the topical applications are considered as safe unless a person has allergic reaction to the plant itself or the herbal is not applied correctly and cause adverse reactions [3].

To improve topical effectiveness of the biologically active ingredients, researchers worldwide test the possibility of integrating aloe vera extract into nanofibers [4,5].

Electrospinning equipment with a syringe is the most common device mentioned in the published papers, therefore in this work researchers used syringeless (needleless) electrospinning method to evaluate differences in morphology, mechanical and bioactive possessions of the nanofiber mats obtained by added distinct aloe percentage in spinning solution. Needleless electrospinning compared to the conventional syringe-based system has such advantages as high scalability, productivity, web and fibres diameters uniformity, wide choice of polymers and substrates, as well as economical operation and easy maintenance [6].

2. Materials and methods

Polyvinyl alcohol (PVA; 81365 Aldrich Mowiol® 18-88 Mw ~130,000) and liquid aloe extract (AV) obtained from local shop “BB Factory Cosmetics” were used to prepare spinning solutions in different concentrations (Table 1.).
Table 1. Samples of prepared polyvinyl alcohol (PVA) and aloe vera liquid extract (AV) spinning solutions

| Composition                  | AV content wt% |
|------------------------------|----------------|
| PVA (10 wt%)                 | 0              |
| PVA (10 wt%) and AV          | 3; 5; 10; 15   |

PVA and AV were dissolved in distilled water using Biosan magnetic stirrer with hot plate MSH-300. PVA was stirred (1000 rpm) for 2 hours at 120-135 °C and then cooled down to 20°C. AV was stirred (1000 rpm) for 30 minutes without heating in order not to impact bioactive compounds.

Electroconductivity and viscosity were measured before electrospinning as the both values show the possibility of obtaining nanofibers before the production. It is known that for the solution to be spinnable, viscosity value must be 100-2000 millipascal-seconds (mPa-s) [6]. Electroconductivity was measured using universal conductivity meter Greisinger GLF 100.

Nanofiber mats were obtained using Nanospider™ LAB 200 (Elmarco, Czech Republic) with following settings- distance between electrodes- 17 cm, roller speed- 3 rpm, applied voltage- 65 kV, temperature- 20 C°. It is important to set appropriate electrode rotation speed to ensure maximum production rate [7] and distance between electrodes for the solvent to evaporate [8].

Morphology of the nanofiber mats was evaluated and fibers diameters measured on atomic force microscope Dimension Edge Veeco (Bruker, USA) with silicone cantilever OTESPA-R3, f0=300kHz, k=26N/m in tapping mode.

Fourier Transform Infrared (FTIR) spectroscopy analysis was performed on Thermo Scientific Nicolet 6700 spectrometer to characterize electrospun mats.

Tensile strength and elongation at break were tested on Zwick Roell Z2020 universal testing machine.

3. Results and discussion

3.1. Viscosity and conductivity.

Prior to preparing compositions, PVA 10wt% and undiluted AV were measured. Viscosity (mPa-s) of PVA 10 wt% was 411, AV- 0.7, conductivity (µS/cm) of PVA 10 wt% was 390, AV 1084. As shown in Figure 1., viscosity decreases (by 22 %) and conductivity 1.8 times rises when AV concentration in the solution is increased from 0 to 10 wt%.

![Figure 1. PVA and AV spinning solution properties](image-url)
Authors concluded that the ratio of conductivity 942 μS/cm and viscosity 395 mPas·s does not maintain the stability of production process as AV 15 wt% and PVA solution did not form quality web.

Growth of conductivity in AV range under investigation could be described by linear equation (1) and increase of viscosity by nonlinear equation (2).

\[
Y_C = 41.40 x + 540.22 \quad (1)
\]
\[
Y_V = -1.22 x^2 + 3.49 x + 394.46 \quad (2)
\]

3.2. Morphology of nanofiber mats
Morphology of 10 wt% PVA and 3 wt%, 5 wt% and 10 wt% AV is shown in Figure 2. As AFM micrographs show, fibers are well defined. As AV concentration increases, fibers show a slight tendency to become finer.

Figure 2. Morphology of nanofiber mats- a (PVA+3% AV), b (PVA+5% AV), c (PVA+10% AV)

3.3. Diameters of nanofibers
Fiber diameters were measured from AFM images. 100 diameter measurements of each sample were taken to ensure data reliability, relative error do not exceed 3.5%. In medical applications fibres diameters are important parameters in drug release process [9].

Figure 3. Average and median diameters of nanofibers
As AV concentration rises, diameter values decrease (Figure 3), average and median values are very similar which means that diameters distributions are symmetrical. It is seen from graphs that AV 3wt% is too small concentration to influence fibres diameters. The average diameters drop down by 10 % when AV concentration increase to 5 wt %. Decrease of diameters are moderate in a range of AV concentration increase from 5 to 10 wt%.

While average diameter size of syringe electrospun nanofibers were 123 nm (10% PVA and 5% AV powder) [10], 10% PVA and 5 wt% AV composite nanofibers obtained by needleless electrospinning have average value of 692 nm.

3.4. Fourier transform infrared (FTIR) spectroscopy
FTIR spectra of aloe vera values are in wave length range of 330.69 cm\(^{-1}\) to 3398.3 cm\(^{-1}\) and the chosen preparation method can either sustain or damage bioactive compounds of the plant [11].

In this study FTIR measurements show (Figure 4.) that there are three new peaks (921, 922.12 and 923.67) in PVA and AV composites compared to pure PVA 10 wt% sample. The results confirm that AV is successfully integrated into nanofiber mats.

![Figure 4. Fourier Transform Infrared (FTIR) spectroscopy](image)

3.5. Mechanical properties of nanofiber mats
Tensile strength and elongation at break are techniques to characterise mechanical properties of nanofibers and nanofiber mats. It is suggested to place fabricated samples on a frame or paper (Figure 5.) to avoid the breakage or slippage of the fibers at the grips and misalignment of the sample as mechanical grippers of the tested device are not suitable for nanofibers [12].
Figure 5. Preparation of a) a single nanofiber for tensile testing in Chatillon tensile tester [12] and b) nanofiber mat sample in Instron Universal Tester (Model 2519-107) [13]

Aloe vera is widely used in nanofibrous structures for skin substitutes nowadays [14], therefore produced materials should have excellent mechanical properties when applied on skin to guarantee comfort while delivering active ingredients to cells.

As shown in Figure 6 and equations (3) and (4) tensile strength of web increases nonlinear with the increase AV content. At max investigated AV concentration (PVA 10wt %/AV 10 wt %) tensile strength of nanofiber mats increase by 210 % and elongation at break by 80%.

Figure 6. Mechanical properties of nanofiber mats

\[ Y_S = 1.25 \times x^2 - 0.82 \times x + 22 \]  \hspace{1cm} (3)
\[ Y_E = 4.57 \times x^2 - 6.94 \times x + 63.04 \]  \hspace{1cm} (4)

Where: \( Y_S \) and \( Y_E \) tensile strength and elongation at break, \( x \) - AV concentration, %.

4. Conclusion
The results of this study show that AV in concentrations 3, 5 and 10 wt% added to 10 % PVA solution can be electrospun by roller electrospinning method, but 15 wt% AV does not form quality nanofiber mat.
AV influences viscosity and electrical conductivity of the spinning solution, diameters of nanofibers and morphology and mechanical properties of nanofiber mats.

AV decreases viscosity and increases conductivity of the spinning solution. Nanofiber diameters tend to decrease when AV concentration is increased.

FTIR spectroscopy analysis confirmed that AV was successfully integrated into PVA nanofiber mat. Mechanical test results show that AV enhances tensile strength and elongation at break values of PVA nanofiber mat.

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