Electrospinning of Chitosan/Poly (Vinyl Alcohol) Nanofibrous Membranes as an Alternative Wound Dressing Material

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ELECTROSPINNING OF CHITOSAN/POLY (VINYL ALCOHOL) NANOFIBROUS MEMBRANES AS AN ALTERNATIVE WOUND DRESSING MATERIAL

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Abstract - A study of the influence of CS concentrations on the tensile properties corresponding to the fiber morphology formed in CS/PVA membranes is this research focus. The ability of the developed membranes for an alternative wound dressing material is also studied. The CS/PVA membranes were prepared using chitosan (CS) solution with different concentrations of (1, 3, 5, and 7 wt. %) combined with 10 wt. % poly (vinyl alcohol) (PVA) and a CS/PVA ratio of 5/95 by electro spinning method. The membranes with an optimized CS concentration of 3% and different CS/PVA ratios of 10/90, 15/85, and 20/80 were also studied. The membrane with 3 % CS concentration showed an optimum tensile strength (5.62 ± 0.47 MPa) with a CS/PVA ratio of 5:95. The improvement of the CS/PVA ratio increased the tensile strength and modulus of the membranes, which reached a maximum value (7.68 ± 1.94 MPa) at a CS/PVA ratio of 20:80, leading to a high density of tight bonds between the fibers. A simulation by finite element method (FEM) on the tensile properties of the CS/PVA membrane has shown that the membrane can be used as an alternative wound dressing material.

Keywords - Chitosan, PVA, Nanofibrous Membrane, Tensile Properties, Wound Dressing Material

I. INTRODUCTION

In the design of materials for some industrial applications, the achievement of the mechanical properties of the material, which is matching with the production target is one of the required product specifications. For example, the tensile properties of nanofiber membranes that are useful in certain applications such as food packaging and biomedical, should at least have a high strength and elasticity of the products. The rapid growth of nanofiber technology is the result of the nanotechnology revolution, which has made it possible to produce membranes from polymers. In this case, electro spinning is a simple method and useful for making polymer nanofibers. The simplicity of process technology in producing fiber in size from nanometer to sub-micro with a non-woven has made the preferred method. Related to the product of nanofiber membranes, a natural polymer of chitosan (CS) and a synthetic polymer of poly (vinyl alcohol) (PVA) were used as base materials in the current study. It is well known that chitosan produced from shrimp shell waste [1–3], is abundantly available in the earth, especially in the regions located near the sea. The effort to make efficient use of the waste is one of the environmental awareness.

In addition, chitosan is very useful in biomedical [4, 5] and food [6, 7] industries due to its advantageous characteristics such as biocompatible, non-toxic, antimicrobial, and biodegradable [8]. On the other hand, PVA is a semi-crystalline synthetic polymer and has almost the same as chitosan, except water solubility. Chitosan is insoluble in water, but soluble in acetic acid (CH_3COOH), whereas PVA, is completely water-soluble, insoluble in the organic solvents and slightly soluble in alcohol [9, 10]. Therefore, a combination of CS and PVA is potentially also used for biomedical and food packaging applications. The studies of CS combined with PVA by electro spinning have been performed to address the characterization of the CS/PVA membrane properties and the use for wound dressing and wound healing [11–14]. The nanofiber membrane of the CS/PVA blend is better in mechanical properties than the neat PVA membrane and may be used in soft tissue engineering. The electrospun membranes made up of 1% chitosan and 10% PVA solutions combined with some different ratios of PVA/CS 80/20, 70/30, 60/40, and 50/50 were potential for wound care. The healing of the wound using the PVA/CS membrane was approximately 40% faster than the open wound [11]. PVA/CS nanofiber membrane tends to be an excellent material for wound dressing, it does not irritate the skin, although it has been in contact with the skin for a long duration (more than a few months) [12].

One % CS and 8% PVA solutions blended at the ratio of 50/50 resulted in a fiber structure closed to the natural tissues, indicating that it is capable of wound healing due to infection [13]. The CS/PVA nanofiber membrane at CS/PVA ratio of 25/75 and an average fiber of about 289 nm was very decisive in healing a diabetic wound on the mice [14]. However, few studies discussed the characterization of the tensile properties of CS/PVA nanofibrous membranes and the use of measurement properties for stress analysis. For wound dressing application, the mechanical property is one of the significant properties of the membrane. The membrane should have high strength,

sufficient modulus elasticity, and high strain, such as the properties of the natural tissue. When membranes are used, membranes may accept tensile loads. For example, if the membrane is used to cover the injury that occurred to the elbow or knee, and then the hand or legs are moved from a straight to a bent position, the membranes will receive a tensile load. It is, therefore, necessary to make the data of the tensile properties of the membranes and to perform the characterization and analysis. The current study discussed the tensile properties of the CS/PVA membranes affecting by the CS concentrations associated with the fiber morphology formed in the nanofibrous membranes. Besides, the capability of the membranes for alternative wound dressing material based on the data on tensile properties data was studied by a simulation using the finite element method (FEM) supported by Abaqus Simulia software. This analysis is required as the first step of confirmation before the membrane is produced for a commercial product.

II. DETAILS EXPERIMENTAL

2.1. Materials and Preparation of CS/PVA blend

Chitosan (CS) micro-powder and acetic acid (glacial, 99 – 100%) were obtained from Sigma Aldrich and Merck, respectively. PVA (Gohsenol, Mw: 22,000 g/mol) was purchased from CV. Multi Kimia, Yogyakarta, Indonesia. Chitosan (CS) solutions with varying concentrations of 1, 3, 5, and 7 % (w/w) were prepared by dissolving chitosan micro-powder in the 2% (w/w) acetic acid solution. The dilution of the acetic acid solution was carried out at room temperature by stirring the solution at 200 rpm for 15 minutes. Subsequently, the dissolution of chitosan powder in the acetic acid solution was performed by heating the chitosan solution at around 80°C and magnetic stirring at 200 rpm for about 45 minutes and then cooling down to room temperature. In addition, the PVA solution was ready at a concentration of 10% (w/w).

2.2. Manufacture of the CS/PVA nanofiber membranes

The mixture of CS and PVA solutions used for the spinning solution was prepared in two types of CS/PVA blends, namely (1) CS/PVA blends at a CS/PVA ratio of 5/95 with 1, 3, 5 and 7 % CS concentrations, and (2) CS/PVA blends at 3% CS concentration with CS/PVA ratios of 5/95, 10/90, 15/85 and 20/80. The viscosity of all CS/PVA blends was measured with a Brookfield viscometer. All CS/PVA blends were inserted into the syringe in the electro spinning machine for the production of CS/PVA nanofiber membranes. The electro spinning process ran at an applied voltage of 18 kV, a fixed distance between the tip and the collector plate of 16.5 cm, a needle diameter of 0.6 mm, and a feed rate of ~0.5 ml/h.

2.3. Characterization

Scanning electron microscopy (SEM, Hitachi 3500-SU) was used to characterize the fiber morphologies of the produced membranes. The fiber diameter measurement was carried out by measuring at least 100 fibers on the SEM image using the ImageJ open-source software. The tensile test for all the membranes was carried out according to ASTM D-882, using a testing machine (Zwick Z0.5 Germany) at a crosshead speed of 10 mm/min and a gauge length of 20 mm. Seven membrane specimens with an average thickness of approximately 40 μm were prepared for each variation, in which the thickness was measured under an optical microscope (Olympus SZ 61).

2.4. Simulation

The simulation used the tensile properties of the second round of CS/PVA blending membranes. The membrane size of this simulation was modeled on the basis of a large-scale commercial band-aid: i.e., 60 mm in length and 30 mm in width. The use of the membrane in this research is to cover the wound near the elbow. When the position of the membrane moves from a straight position to be a bent position, the displacement will be occurred. This process is simulated and analyzed using the Abaqus Simulia software-supported FEM.

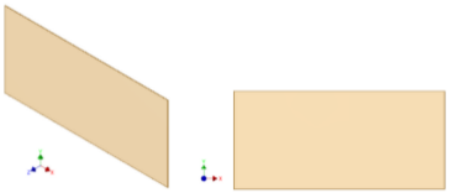

Fig.1. Geometric of the membrane.

In this simulation, the geometric of the membrane was firstly made (Fig.1). The geometric was then imported to the finite element-based software and given a force until the value of von misses is equivalent to the tensile strength of the used membrane.

III. RESULTS AND DISCUSSION

3.1. Optimization of CS Concentration

In the study of CS/PVA blend by electro spinning, the use of CS concentrations is different from each other. The CS concentration used in this study was optimized based on the result of

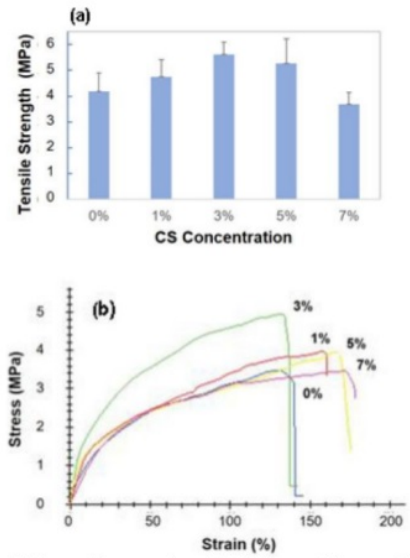

Fig.2. The tensile properties of CS/PVA nanofiber membrane at CS/PVA ratio of 5/95. (a) tensile strength vs CS concentration, and (b) stress-strain curve.

The tensile properties (Fig.2). In this case, the CS/PVA membrane with a concentration of 3% CS shows an optimum tensile strength value, whereas its elongation is almost the same as the elongation of the neat PVA. The elongation at break ranging from 100% to 150% seemed to be a part of the range of natural tissue properties. As a result, 3% of CS concentration was used to better classify CS/PVA mixtures by varying CS/PVA ratios. A study of the CS/PVA nanofibrous membrane with 3% CS concentration has achieved an optimum value of CS/PVA ratio at 50/50 based on better fiber structure formulation compared to other ratios [15]. Another research study varied the CS concentration from 0% to 3% to understand its effects on the formation of fiber during the electro spinning process. This research confirmed that the CS concentration of less than 2% and higher than 3% made the solution is not spinnable due to excessive viscosity of the solution [16- 17].

3.2. FTIR Analysis

The chemical structure of pure PVA, chitosan, and CS/PVA has different characteristics (Fig.3). The FTIR spectrum of pure PVA exhibits the main positions related to –OH stretching (3364 cm^{-1}), –CH stretching (2931 cm^{-1}), stretching vibration of C=O groups (1735 cm^{-1}), –CH bending (1427 cm^{-1}), –C–O– stretching (1087 cm^{-1}) [18, 19]. In the spectrum of pure chitosan, the peaks at 3425 cm^{-1} and 2900 cm^{-1} are correspond to –OH and $–NH_2$ stretching groups and –CH stretching vibrations [18, 20]. The absorption bands identified at 1643 cm^{-1}, 1580 cm^{-1} and 1087 cm^{-1} are related to amide I, II, and a weak amide I, respectively [21], while those positioned at 1087 cm^{-1} and 887 cm^{-1} are associated with –C–O– stretching vibration [18]. The broad peak corresponding to –OH stretching at approximately 3400 cm^{-1} is present in all spectra, which sifts to lower wave number as PVA content increases [19]. In the CS/PVA spectra, the coexistence of the functional groups of the C=O groups, amide I, and II is represented in the CS/PVA (20/80) spectrum: i.e., at the positions of 1728 cm^{-1}, 1651 cm^{-1}, and 1566 cm^{-1}, respectively. Such results showed strong compatibility between CS and PVA in the CS/PVA nanofiber membranes. In the event that they are incompatible, the spectrum will show to be similar to the pure component one [19].

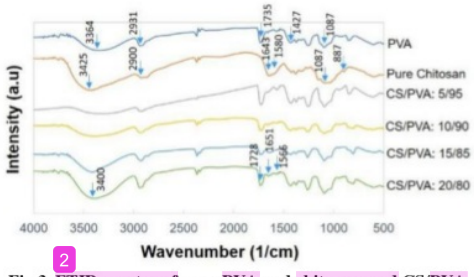

Fig.3. FTIR spectra of pure PVA and chitosan, and CS/PVA nanofibrous membranes at 3% CS concentration with various CS/PVA ratios.

3.3. Fiber morphology

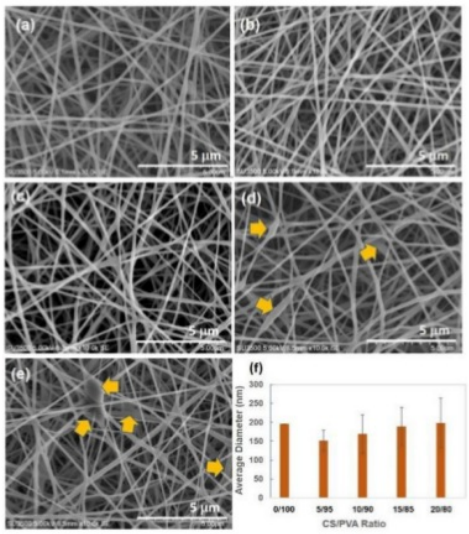

Fig.4. SEM images of nanofibrous membranes of CS/PVA blend with various CS/PVA ratios; 0/100 (a), 5/95 (b), 10/90 (c), 15/85 (d), 20/80 (e) showing the formation of beads in (d) and (e) (see arrows).

The fiber morphologies formed in all membrane specimens reveal straight-oriented fibers with a uniform size distribution, particularly at SC/PVA

ratio of 0/100 (neat-PVA) (Fig.4a) and 5/95 (Fig.4b). The higher the CS content, the larger the average fiber diameter (Fig.4f), which corresponds to the viscosity of CS-PVA spinning solutions (Table 1), which increases by increasing the CS concentration. The average fiber diameter of all CS/PVA fibrous membranes is in the range of 100 - 200 nm, which is proportional to a previous result [15]. Enhancing the viscosity of the spinning solution is not only providing an impact on the fiber diameter but also the formation of beads as fiber-defect, that has gradually been produced at CS/PVA ratio of more than 5/95 (Fig. 4d and 4e, see arrows). In this case, viscosity is a function of intermolecular interaction in the polymer structure. Thus, the viscosity of the solution has a significant impact on

Specimen at CS/PVA	Viscosity (cP)	Average fiber diameter (nm)
0/100	340	196
5/95	465	151
10/90	554	169
15/85	606	195
20/80	809	198

Table 1: Viscosity of the spinning solution of CS-PVA

the formation of fiber other than surface tension and electrical conductivity [22]. However, the beads formed in these membranes do not obstruct the cross linking of the fiber. It should be noted, therefore, that 3% of CS concentration yielded the appropriate viscosity of the solution resulting in good electro-spinnability.

3.4. Tensile properties related to fiber morphologies

The fiber morphology in terms of fiber structure affects the tensile properties of the nanofibrous membranes. As far as our experience in the manufacture and characterization of the electro spun membranes is concerned, the fiber crosslink has positively played a crucial role in the tensile properties of the membranes. The higher the volume fraction of crosslink between fibers in the membrane, was formed, the higher the tensile strength of the membrane. Based on the present result (Fig.5a), the addition of chitosan gradually improved the tensile strength of the CS/PVA membranes, but the elongation varies (Fig.5b). The earlier result [15], which used around 2.5% CS concentration and different CS/PVA ratios showed comparable tensile strength (6.13 ± 0.72 MPa), significantly higher tensile modulus (94.2 ± 10.2 MPa) and lower elongation at break (14.13 ± 1.25%) at CS/PVA ratio of 15/85 compared to the present results. These measures were higher than the properties of CS/PVA membranes at CS/PVA ratios of 25/75 and 30/70, explaining that an increase of chitosan content yielded unfavorable tensile properties, especially for wound dressing material due to too high modulus elasticity. Compared to another study [21], without the addition of chemical cross linking (tetraethyl ortho silicate/TEOS), these present results showed higher specifically in the tensile strength, but lower in the elongation at break. Besides, these tensile properties are also better than the other [23], although the chemical cross linking of glutaraldehyde (GA) has been used. In principle, the nanofibrous membrane with very high modulus elasticity is not applicable for wound dressing material. The tensile properties of these nanofiber membranes are used for the simulation analysis using Abaqus Simulia software in the next paragraph to ensure whether the produced membranes would be applicable for an alternative wound dressing material or not. Based on the result in Fig. 5a, the CS/PVA membrane with a CS/PVA ratio of 20/80 reaches the highest tensile strength (7.68 ± 1.94 MPa). This property was used for the following simulation.

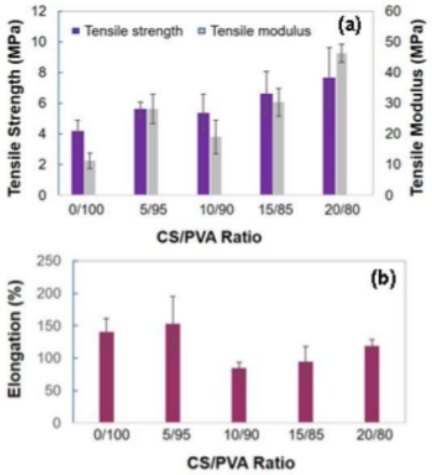

Fig.5. Tensile strength and modulus vs CS/PVA ratio (a) and elongation vs CS/PVA ratio (b).

3.5. A simulation result applied to the CS/PVA nanofiber membrane

The CS/PVA membrane with a CS/PVA ratio of 20/80 is selected for this simulation due to its highest tensile strength. The CS/PVA membrane application model in Fig. 6. displays the length of the membrane at a straight position of 60 mm. However, at a bent position, the range extends to be 65 mm, explaining that there is a displacement of about 5 mm toward x-direction. In this case, the trial and error were performed to determine the thickness of the membrane. The membranes with the dimension of 60 mm length, 30 mm width and various thicknesses from 40 μm to higher than 100 μm have been tried for modeling from a straight to the bent position. The results showed that the membrane had failed due to a thickness of less than 110 μm. Nevertheless, at an average thickness of 110 μm, when determined by an

optical microscope (Fig.7), the membrane worked excellently with a displacement of about 5 mm, as depicted in Fig.6. The membrane with a dimension of 60 (l) x 30 (w) x 0.11 (t) is therefore used for stress analysis.

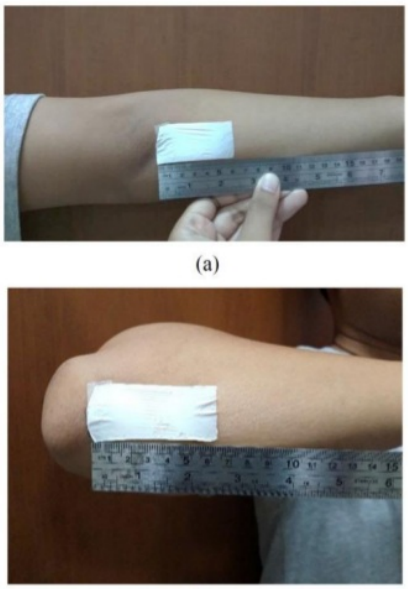

Fig.6. An application model of the CS/PVA membrane to cover the wound in a straight position (a) and in a bent position in which the displacement occurred.

The stress analysis of the membrane performed using Abaqus Simulia software shows the geometrically based-simulation result (Fig.8) and von misses stress of the membranes when the elbow of a hand is folded. It is shown that the maximum von misses of the membrane is about 7.67 MPa, which is equivalent to the maximum tensile strength, and the displacement demonstrates a maximum value of around 5 mm, which corresponds to the actual measurement exhibited in Fig.6b.

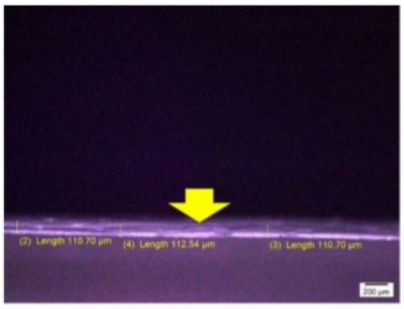

Fig.7. An optical micrograph of a cross-section of the membrane showing an average thickness.

This result suggested that the CS/PVA membrane with a CS/PVA ratio of 20/80 would be useful for an alternative wound dressing material by designing a dimension of 60 mm in length, 30 mm in width and a minimum thickness of 0.11 mm (110 m) which provides a 5 mm displacement of the membrane. However, a significant improvement in the tensile properties of the nanofibrous membrane is required in the next research.

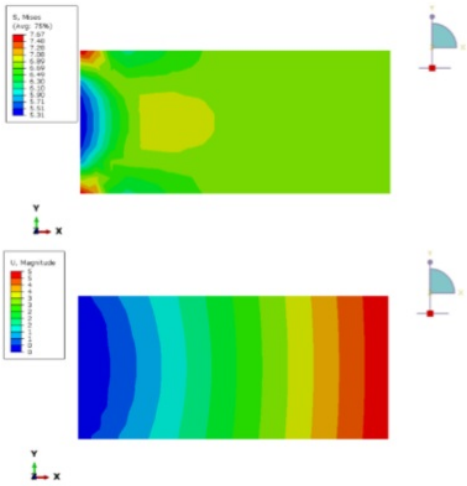

Fig.8. Abaqus Simulia simulation result.

IV. CONCLUSION

The electrospinning technique has successfully manufactured the CS/PVA nanofibrous membranes. The influence of the variation of CS concentrations and CS/PVA ratio on the tensile properties of CS/PVA membranes has resulted in some significant points. Increased CS concentration increases the tensile strength of the CS/PVA membrane with a CS/PVA ratio of 5/95, resulting in an optimum CS concentration of 3% with a tensile strength of 5.62 ± 0.47 MPa. Various CS/PVA ratios at 3% CS concentration showed a gradual increase in the tensile strength and modulus in which the maximum values reached by the membrane with CS/PVA ratio of 20/80 were 7.68 ± 1.94 MPa and 46.3 ± 2.89 MPa, respectively, and the elongation at break of 118.65 ± 10 %. Based on the simulation result using FEM supported by Abaqus Simulia software, the CS/PVA membrane with those properties is recommended as a wound dressing material by designing a dimension of 60 mm in length, 30 mm in width, and a minimum thickness of 0.11 mm (110 μm).

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