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MECHANICAL PROPERTIES OF PVA/ALGINATE MEMBRANES FABRICATED USING ELECTROSPINNING AS A WOUND DRESSING

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ABSTRACT

Alginate is an interesting natural biopolymer due to its many benefits and good biological properties. Observing the mechanical properties of PVA/Alginate fibers made by electrospinning machines can help predict their behavior and measure their performance under various conditions including applied external forces. Therefore, this study investigated the elastic properties of PVA/Alginate membranes, specifically tensile strength and elongation. The fabrication material used is PVA / Alginate solution with a PVA solution concentration of 20% and alginate solution 2.5%. The electrospinning process is carried out by optimizing at a voltage of 25 kV, the distance between the spinneret end and the collector is 15 cm, the flow rate is 130, and the spinneret diameter variations are 0.4 mm, 0.6 mm, and 0.8 mm. To determine the morphology of the fiber surface, observations were made using scanning electron microscope (SEM). From this observation, it is known that the morphology formed using a 0.4 mm spinneret has the tightest structure among the three sizes of spinneret diameter. To determine the effect of spinneret diameter on the value of tensile strength and elongation, tensile strength tests are carried out. The tensile strength and elongation values of the membrane obtained with variations in spinneret diameters of 0.4 mm, 0.6 mm, and 0.8 mm are 1.96 MPa, 3.77 MPa and 6.11 MPa respectively and the elongation values are 17%, 52.7%, and 97%. With medical material standards, it has a tensile strength value between 1 MPa – 24 MPa and an elongation value between 17% – 207%, so that PVA/Alginate fiber membranes have the potential to be applied as wound dressings.

Keywords: alginate; electrospinning; mechanical properties; wound dressings

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INTRODUCTION

In recent years, the development of elektrospinning techniques has attracted great attention in producing polymer fiber membranes with high surface-to-volume ratios and diameters ranging from microns to several nanometers [1],[2],[3],[4]. This electrospinning technology has been used to make fiber membranes from various synthetic or natural polymers. Natural polymers are chosen because they have high biocompatibility and biodegradation benefits and can be used in biomedical applications. However, problems often arise in the formation of fiber membranes from these natural polymers [5]. Therefore, to overcome these problems, mixing natural polymers with synthetic polymers is carried out in the manufacture of fiber membranes.

Alginate is a type of naturally derived polysaccharide that is biocompatible and has many benefits. Alginate belongs to a family of polysaccharides derived from the cell walls of brown seaweed. Sodium Alginate is a natural alginate formation with non-toxic, biodegradableand biocompatibleproperties. Alginate is widely developed as a source of biomedical ingredients, including wound dressings [6], drug carrier [7] tissue engineering scaffold [8]. In its performance, sodium alginate cannot stand alone, but must be mixed with synthetic polymer solutions, so that there is a hydrogen bond interaction between two or more mixed polymers. Poly (vinyl alcohol) is a synthetic polymer used to help the formation of fibers in alginate during the electrospinning process. Poly (vinyl alcohol) can improve intermolecular spinability in the electrospinning process [9],[10].

The mechanical properties of fiber membranes have an influence on the strength and ability of electrospinning materials. Ability to withstand forces and loads measured based on material response [11]. There are several factors that affect the mechanical properties of the membrane resulting from elektrospinning, namely morphology, diameter and crystallinity of fibers. Tensile strength and elongation are mechanical properties of a material. Tensile strength and elongation indicate physical quantities that describe the depth of mechanical properties of the fiber membrane [12].

In the study Robaitullah. (2017), PVA/nanochitosan nanofiber membrane has been made by electrospinning method to determine the mechanical properties and morphology of nanofiber membranes[13]. In the research of Subyakto et al. (2009), the smaller diameter of the fiber makes a material have a higher modulus of elasticity and tensile strength. The increase occurs because the ratio between the diameter and length of the fiber becomes smaller [14]. In the study of Stachewich et al. (2012), related to the formation of PVA fiber membranes which showed an increase in modulus of elasticity along with a decrease in fiber diameter [15].

Previous studies have reviewed the effect of spinneret size on mechanical properties, but in that study only used poly (vinyl alcohol) solution [11]. In this study, a review of the effect of spinneret size on mechanical properties including tensile strength and elongation using poly (vinyl alcohol) solution with the addition of alginate as filler. The values of tensile strength and elongation are obtained by conducting tests using tensile strength testing equipment. To determine the morphology of the surface, observations were also made using scanning electron microscope (SEM). The results stated that the larger the diameter of the spinneret used, the greater the value of tensile strength and elongation possessed by the fiber membrane.

METHODS

This research begins with preparing tools and materials. Materials used in this study include PVA, alginate, alcohol and distilled water. The equipment used in this study included electrospinning machines, preparation glass, hot magnetic stirrer, and testing equipment.

Making Alginate Solution

The preparation of alginate solution begins by mixing alginate powder with distilled water according to a predetermined concentration of 2.5%. Then the alginate powder is put in distilled water in beaker glasses with a temperature of 70^{o}C for 20 minutes at a stirrer speed of 900 rpm for the homogenization process of the alginate solution. After that, the alginate solution is allowed to stand until the temperature of the solution is equal to room temperature.

Preparation of Poly (vinyl Alcohol) Solution

Making PVA solution begins by mixing PVA powder with distilled water according to a predetermined concentration of 20%. Mixing is done little by little after the distilled water are heated and is characterized by the presence of moisture around the walls of the beaker glass. Then the mixing process is heated with a temperature of 90°C for 1 hour with a steering speed of 900 rpm. After that, the solution is allowed to stand until the temperature of the solution is equal to room temperature.

Preparation of PVA/Alginat Solution

The preparation of PVA/Alginate solution begins by mixing PVA solution and alginate solution each with a volume of 9: 1. Then the solution is heated to a temperature of 80°C for 15 minutes at a stirrer speed of 800 rpm. After the homogeneous solution is then poured into the syringe pump and allowed to stand until the solution is room temperature. To reduce bubbles in the solution due to stirring, the syringe pump is placed vertically and the lid is opened, this is used to remove air in the syringe pump.

Electrospinning Optimization

The process of optimizing electrospinning begins by preparing PVA/Alginate solution that has been inserted into the syringe pump. Running by setting several fixed parameters that affect the electrospinning process include a voltage of 25 kV, a spinneret tip distance to a collector of 15 cm, a flow rate of 130, and variations in spinneret diameters of 0.4 mm, 0.6 mm and 0.8 mm with a time of 1.5 hours. Furthermore, a syringe pump containing the solution is installed on the electrospinning machine until the running process is complete.

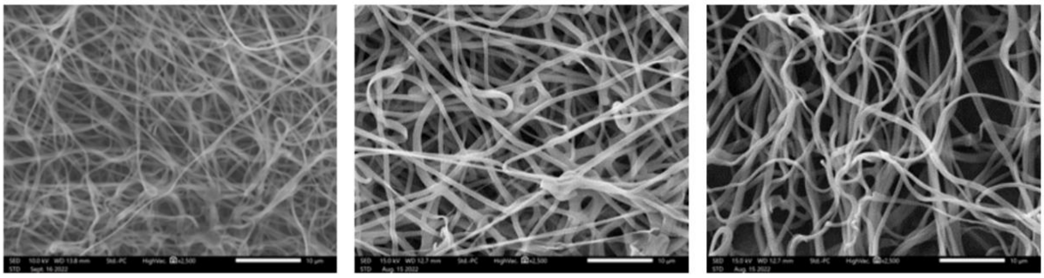
Sample morphology

The morphology of nanofiber samples was performed using scanning electron microscope (SEM). SEM (Scanning Electron Microscope) is used to determine the surface morphology and diameter size of the resulting fiber and to determine the condition of the fiber formed. In determining the average diameter of fiber using the ImageJ and OriginPro applications.

Mechanical Test

Mechanical testing is used to determine the value of tensile strength and elongation of fiber membranes. The test was carried out using a tensile strength test tool, Brookfield CT3. The testing process is drawn until it obtains the amount of force that can be withstood by the sample. By knowing the tensile strength and elongation, in its application it can be known that fiber membranes with PVA/Alginate materials can be used in wound dressing applications and so on.

RESULTS AND DISCUSSION



(a) (b) (c)

Figure 1. PVA/Alginate fiber membrane surface structure SEM results with variation in diameter of spinneret with electrospun PVA fiber a)0.4 mm, b)0.6 mm, and c)0.8 mm

SEM (Scanning Electron Microscope) is used to determine the surface morphology and diameter size of the resulting fiber and to determine the condition of the fiber formed. Figure 1 (a) shows a fiber membrane resulting from a spinneret with a diameter of 0.4 mm. The resulting fibers are more uniform, continuous and separate from each other. This shows that fibers formed using PVA/Alginate with a PVA solution concentration of 20% and alginate solution 2.5% with voltage parameters of 25 kV, spinneret diameter 0.4 mm, spinneret tip distance to collector 15 cm and flowrate 130 can produce a good fiber membrane.

The average diameter of the fiber was also measured using the ImageJ and OriginPro applications. Diameter fiber produced using a spinneret with a diameter of 0.4 mm, which is 378 ± 27 nm. Figure 1 (b) shows the fiber produced from a spinneret 0.6 mm in diameter. The fibers formed are uniform, continuous and many are fused with each other. This can happen because there are still bubbles in the solution so that the polymer with drawal process is not optimal [16]. By using a spinneret with a diameter of 0.6 mm, the average diameter of the fiber produced is 498 ± 36 nm.

Figure 1 (c) shows the fiber produced from a spinneret 0.8 mm in diameter. By observing the picture, it is obtained that the fibers formed are quite continuous and quite uniform according to their size. This occurs because of the instability of conditions when withdrawing polymers from the spinneret to the collector, which causes the evaporation of the solution to differ so that the fibers that reach the collector are not uniform. After measurements were made using ImageJ and OriginPro, the average diameter size on a spinneret with a diameter of 0.8 mm was 504 ± 12 nm. This shows that the smaller the diameter of the spinneret used, the smaller the diameter of the fiber produced. This is in accordance with what was stated by Hochleitner et al. (2014) that a lower spinning needle diameter will result in a smaller fiber diameter as well[17].

In addition to the size of the fiber produced, mechanical properties such as elongation and tensile strength also need to be considered for its application. Here are the values of tensile strength and elongation resulting from the formed membrane.

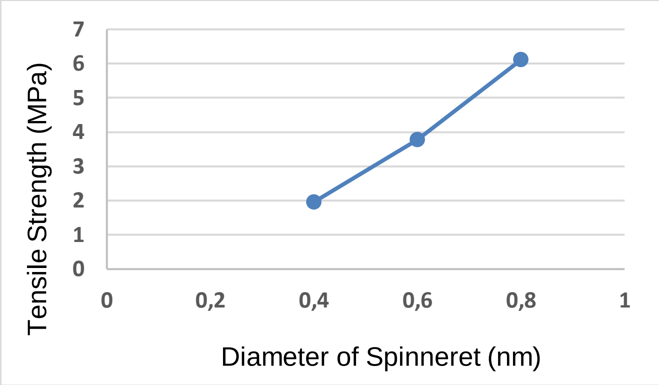


Figure 2. Plot of the variation in diameter of spinneret with electrospun PVA fiber tensile strength

In Figure 2 it can be observed that the fiber membrane formedwith a spinneret with a diameter of 0.4 mm produces a tensile strength value of 1.96 MPa. The fiber membrane formed with a spinneret with a diameter of 0.6 mm has increased with a tensile strength value of 3.77 MPa and the fiber membrane with a spinneret with a diameter of 0.8 mm has the highest tensile strength value of 6.11 MPa. This is due to the large difference in force that can be with stood by each fiber and the deformation that occurs so that the tensile strength value becomes higher. This happens because mixing PVA with alginate forms hydrogen bonds, namely the H element in PVA binds to the O element in alginate. The existence of these bonds can increase intermolecular spinability and mechanical properties of the fiber membrane formed [9],[10].

Polyvinyl alcohol has water-loving properties (hydrophilic), so it can dissolve when interacting with water because of the active group it has, namely the OH group through hydrogen bonding [18]. PVA has high tensile strength and flexibility, but when humidity is high, PVA will absorb more water. Water serves as an adhesive that will reduce tensile strength, but can increase elongation and tear strength [19]. PVA has been widely used in the formation of nanofiber membranes using electrospinning method. PVA is also used as a matrix for other polymers to improve their mechanical properties [20].

Based on Annaidh et al. [21], medical material standards have tensile strength values between 1 MPa – 24 MPa. With this standard, it can be seen that the fiber membrane produced with changes in the diameter of the spinneret used has met the standard, namely >1 [21].

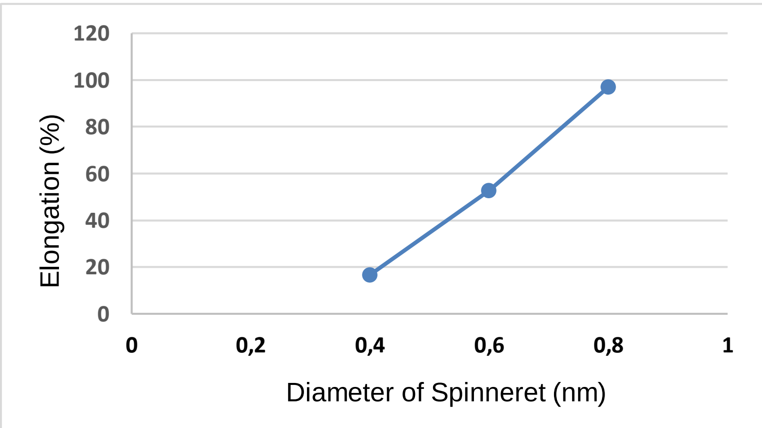


Figure 3. Plot of the variation in diameter of spinneret with electrospun PVA fiber elongation

Figure 3 shows that the larger the spinneret diameter, the elongation value tends to increase. The spinneret fiber membrane has a diameter of 0.4 mm, has an elongation value of 17%. Fiber membrane with fabrication using a spinneret with a diameter of 0.6 mm, the elongation value has increased from the previous one to 52.7%. The increase continues to occur until the fiber membrane results from the use of a 0.8 mm spinneret in the fabrication process, with an elongation value of 97%. This happens because the larger the diameter of the spineret, the smaller the blockage. The nanofibers produced are increasing because the solution that can be drawn by the collector is getting bigger, so the nanofiber membrane is thicker than a spinneret with a small diameter. With a thicker nanofiber membrane, the elongation value will increase

Based on Annaidh et al. [21], medical material standards have an elongation value between 17% - 207%. With this standard, nanofibers formed from changes in spinneret diameter meet the standard for medical material applications, which is >17%.

CONCLUSION

Based on the results obtained, the diameter of the spinneret used in the fiber membrane formation process also affects its mechanical properties. Changes in spinneret diameter affect the value of tensile strength and elongation. The spinneret diameter of 0.4 mm produces a fiber membrane with a tensile strength value of 1.96 MPa and elongation of 16.7%. At a spinneret diameter of 0.6 mm produces a fiber membrane with a tensile strength value of 3.77 MPa, and elongation of 52.7% and for a spinneret with a diameter of 0.8 mm produces a fiber membrane with tensile strength of 6.11 MPa and elongation of 97%. It can be concluded that the larger the diameter of the spinneret, the higher the tensile strength and elongation of the resulting nanofiber. In medical material standards, PVA/Alginate fiber membranes have the potential to be used as wound dressings based on their mechanical properties.

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