Volume 12. Number 1. 11-18 MAY 2023

Jurnal ilmiah pendidikan fisika Al-Biruni
https://ejournal.radenintan.ac.id/index.php/al-biruni/index
DOI: 10.24042/jipfalbiruni.v12i1.14904

P-ISSN: 2303-1832
e-ISSN: 2503-023X

Physicochemical Properties of Characterization of PVP/CA/AE Nanofiber

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Article Info

Article history:

Received: December 11, 2022
Accepted: April 14, 2023
Published: May 24, 2023

Keywords:

Cellulose acetate
Electrospinning
Morphology
Nanotechnology
Polyvinylpyrrolidone

ABSTRACT

Nanofibers are nanotechnologies with cross-sectional diameters ranging from tens to hundreds of nanometers. We explained the definition of nanofibers and their unique physicochemical characteristics. This study aimed to see the characterization of *Polyvinyl pyrrolidone/Cellulose acetate/Aloe vera extract* fiber. The fiber was made with a concentration of PVP/CA in 13% (w/w) and an *Aloe vera* extract in 10% (w/w) using the electrospinning method. The electrospinning technique involves an electrohydrodynamic process in which liquid polymer droplets are electrified to produce jets that elongate to produce fibers. Electrospinning is an easy, fast, and simple technique for producing fibers with sizes ranging from micrometers to nanometers. Process parameters used in this research, such as flow rate of 0,20 ml/hour, high voltage of 12 kV, and drum speed is 200 rpm with the distance to the needle tip of the collector is 75 cm. The result showed that fiber PVP/CA/AE morphology is bead free and homogeneous. The average diameter of PVP/CA/AE fibers was 1115 nm. These results indicate that the resulting fiber is in the form of nanofibers. A small fiber diameter will result in a broader surface area. The XRD result shows that the PVP/CA/AE fibers have a semi-crystal phase.

INTRODUCTION

Scientific disciplines representing a unique combination of natural, mathematical, computer, and materials sciences have proliferated. Nanotechnology is essential because it can investigate and manipulate physical matter on the nanometer scale. (Barkalina et al., 2014). The principle nanotechnology, such as food additives, can be produced to protect food from contamination, thereby enhancing the lifespan of nanomaterial-based lithium-ion batteries (Bhatnagar et al., 2022; Sahoo et al., 2021). On the other hand, nanotechnology has proven to be the most promising agent for faster wound dressing among all the other materials (Bhattacharya et al., 2019). One that is produced through the principle of nanotechnology is nanofiber. Nanofibers have smaller pores and a wider surface than ordinary or conventional fibers (Subbiah et al., 2004). In addition, other advantages of nanofibers compared to conventional fibers are that nanofibers have a very large surface area, are lighter in weight, have a flexible shape, reduce the space required for application, penetrate the optimum performance limits of conventional fibers, and have high economic value (Hulupi & Haryadi, 2018). To produce good nanofibers that contain natural extract, physicochemicals are needed.

The manufacture of nanofibers can be carried out using various techniques, including template synthesis (Chen et al., 2017), phase separation (Huang et al., 2014), self-drafting (Xu et al., 2017), and electrospinning (Ramakrishna et al. 2005). Previously, the manufacture of nanofiber was carried out using a phase separation technique, but the resulting showed less than optimal performance; this was because the membrane pores were still large. Therefore, nowadays, electrospinning techniques are widely used.

Electrospinning is one of the most efficient, cost-effective, versatile, and straightforward methods for fabricating micro- to nano-sized biomimetic nanofibers (Ranjbar-Mohammadi, 2018). The electro-spinning arrangement includes a DC high-voltage power supply, a nozzle spinning connected to a high-voltage DC, a nozzle pump, and a fiber collector (Almafie et al., 2020; Sriyanti & Jauhari, 2019). While supporting components include charge-coupled device (CCD) cameras, monitors, and humidity sensors (Almafie et al., 2020; Yousefzadeh, 2017).

Electrospinning involves (1) Coulomb forces resulting from the applied electrical charge and (2) elongation of the polymer solution. Upon exposure to a very high electrical potential or voltage difference, the charged polymer attracts the collector and forms fiber from nature (Jauhari et al., 2019; Ramakrishna et al., 2005).

Natural polymers and mixtures of balanced polymers and composite polymers have been widely applied in the health sector. For example, a widely used synthetic polymer is *Polyvinyl pyrrolidone*, and a natural polymer is *Cellulose acetate*. Each polymer has advantages such as good permeability, good porosity, biocompatibility, good electro-worthiness, non-toxic, high affinity, low cost, and excellent chemical resistance (Alim Bahmid et al., 2014; Bonan et al., 2015; Khoshnevisan et al., 2018; Kurakula & Rao, 2020). However, natural polymers have the disadvantage that they are easily contaminated with microbes (Mir et al., 2018). However, PVP is easily soluble in water (hydrophilic), reducing the membrane's pore size and permeant (Marbella et al., 2019). PVP is combined with cellulose acetate (CA) polymer to increase water permeability. Cellulose acetate is insoluble in water. To produce a potential nanofiber for wound dressing, the two types of polymer are mixed to extend the degradation period until the wound healing time is complete, improving mechanical properties and maintaining the ability of water vapor and gas permeability (Gizaw et al., 2018)

Aloe vera has the potential to be used in the medical world because of its high antioxidant vitamins, which can improve wound healing (Sosiati et al., 2020). The ingredients contained in *Aloe vera* can be utilized by taking the extract and used in medicine. However, because it is a natural material, it is not durable and needs to be mixed with other polymers.

In this research, we will synthesize nanofibers from PVP/CA/AE using electrospinning technique. Combining natural and synthetic polymers and adding natural extracts are also expected to produce nanofibers with better mechanical properties and contain natural ingredients that can accelerate the wound-healing process. Nanofiber composites have huge potential to be used for tissue engineering and wound dressing (Polini & Yang, 2017). This type of material possesses at least two distinct phases and may act very differently compared to similar materials at a bigger scale. Physicochemical characterization of the developed nanofiber composites is essential to understand their potential fully.

Related research on the synthesis of nanofibers using natural extracts containing antibacterial activity has been carried out, for instance, by Sriyanti et al. (2017) using polyvinylpyrrolidone/cellulose acetate polymer containing Garcinia mangostana L extract, which is used in the medical field as a drug delivery application (Sriyanti et al., 2017). Another research was conducted by Dong et al. (2020) using PVP/Isatis root, which stated that the fibrous structure is well-defined and the surface wetting and permeability characteristics are very good (Dong et al., 2020). however, this study was only limited to looking at the morphology and chemical interactions of the polymer-forming nanofibers; no results showed the nanofiber's crystal structure.

Therefore, this study aims to investigate PVP/CA/AE composite nanofiber physicochemical properties, including morphology, fiber diameter, crystallinity, and chemical interactions, and the result was evaluated.

METHODS

PVP 1,300,000 Mw produced by Sigma Aldrich, cellulose acetate (CA) 30,000 Mw produced by Sigma Aldrich, acetic acid obtained from Brataco Chemical, and *Aloe vera* extract from the local market.

Precursor substances were created by dissolving *Polyvinyl pyrrolidone* and *Cellulose acetate* in acetic acid. PVP/CA solution was prepared with a concentration of 13% (w/w) and stirred for 4 hours at 60^{o}C. Furthermore, *Aloe vera* extract was dissolved in an acetic acid solvent with a concentration of 10% (w/w). After that, put the *Aloe vera* extract into a homogeneous PVP/CA solution with a ratio of 5:3 (w/w) and stir it using a magnetic mixer for 4 hours at 60^{o}C. The precursor solution was put into the syringe to be spun for 7 hours with a flow path parameter of 0,20 / hour, a voltage of 12 kV, a drum speed of 200 rpm, and a distance of 75 cm of the needle tip to the collector.

The morphology of electrospun fiber in PVP/CA/AE solution was characterized using an SEM, the average fiber dimension size was analyzed using an image of J1.52a software, and the dimension of distribution of nanofiber was characterized using Origin pro-2018 software. The average size of crystallization or

crystallinity in PVP/CA/AE was characterized using X-Ray Diffraction (XRD). The research flowchart is shown in Figure 1.

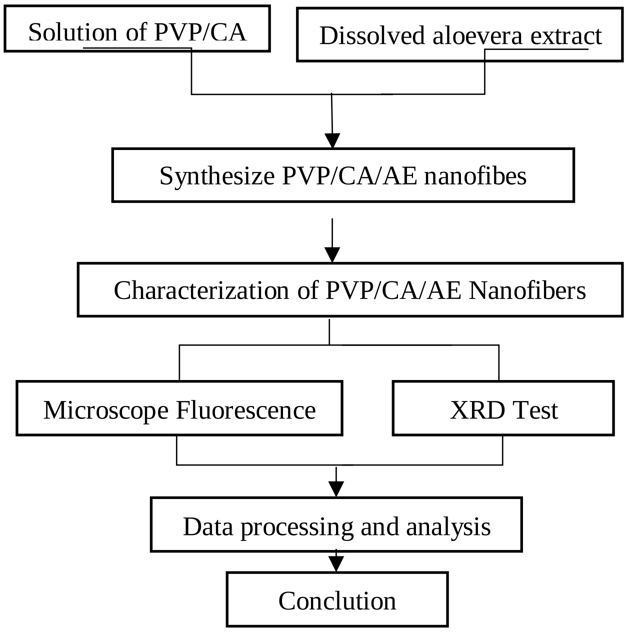

Figure 1. Experiment flowchart

RESULTS AND DISCUSSION

The morphology of PVP-CA and PVP-CA-AE nanofibers is shown in Figure 2.

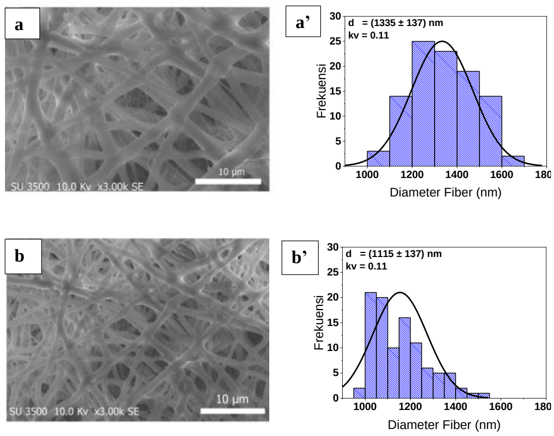

Figure 2. SEM of (a) PVP/CA and (b) PVP/CA/AE; Diameter Distribution of (a') PVP/CA and (b') PVP/CA/AE

The fiber produced by electrospinning is usually in the form of beaded fiber or fine fiber (Kamaruddin et al., 2018). The result fibers in this experiment were fine fibers, such as continuous and long hairs, which were nanometer in size and bead free. Based on the research conducted by (Jauhari et al., 2019), a polymer concentration of 10-15% produces bead-free. The addition of AE to PVP/CA fibers affects their morphology. The addition of *Aloe vera* extract causes the fiber diameter to become smaller. However, the concentration of the solution affects nanofibers' formation. Good solution concentration will produce continuous, homogeneous fibers without beads. Fiber without beads will provide a higher surface area-to-volume ratio and better mechanical properties (Rahma et al., 2016).

Then, the size distribution of PVP/CA and PVP/CA/AE fibers is shown in Figure 2 (a'-b'). The average diameter of the PVP/CA fiber is 1335 nm, the standard deviation is 137 nm, and the coefficient of variation is 0.11. Then, PVP/CA/AE fiber has an average diameter of 1115 nm, a standard deviation of 137 nm, and a coefficient of variation of 0.11. Based on the graph, it can be seen that there is a decrease in the average fiber diameter when *Aloe vera* extract is added to the PVP/CA matrix. This decrease in value is due to the influence of viscosity and conductivity. If the viscosity value is higher, the polymer molecule bond will be stronger (Fatehi & Abbasi, 2020). The higher conductivity of the solution, the more charge accumulates, which causes a stronger pull on the polymer solution so that the elongation is greater. In this study, the addition of *Aloe vera* extract caused a decrease in the viscosity of the solution in the PVP/CA/AE fibers so that the amount of polymer mass to volume decreased and caused the average diameter of the fibers to drop.

Meanwhile, the homogeneity of PVP/CA and PVP/CA/AE fibers can be seen using the fibers' coefficient of variation (Cv). Homogeneous distribution if it has a standard deviation ratio to an average fiber diameter of less than 0.3 (Sriyanti, Agustini, et al., 2020).

Furthermore, crystallinity analysis on PVP/CA/AE nanofibers is shown in Figure 3.

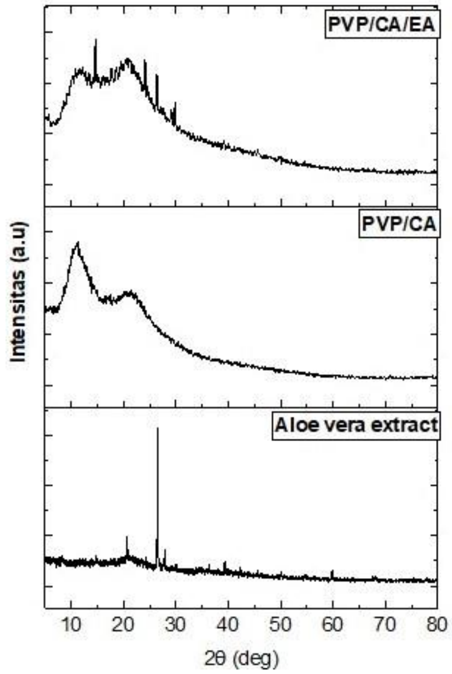

Figure 3. XRD PVP/CA/AE Nanofibers

The XRD patterns of pure AE, PVP/CA nanofiber, and PVP/CA/AE nanofiber composites are shown in Figure 3. The typical AE diffraction pattern has three sharp peaks between the 2θ angles of positions 5° to 80°: 20.68°, 26.56°, and 27.88°. With the sharpest peak at an angle of 26.56°. Based on the peak shape, it shows that *Aloe vera* extract has a crystalline phase. These results align with research conducted by (Aghamohamadi et al., 2018), which stated that XRD *Aloe vera* showed crystalline peaks.

Furthermore, PVP/CA fiber has two weak diffraction peaks between the 2θ angles of 5° to 30° positions: 10.88° and 21.11°. This shows that the PVP/CA diffraction pattern is amorphous. Based on the available literature CA has a semicrystalline phase (Hou et al., 2018), and PVP has an amorphous phase (Sriyanti & Jauhari, 2019). During the electrospinning process on pure polymers, it causes a change from crystalline to amorphous (Dai et al., 2012).

The addition of *Aloe vera* extract resulted in a new peak on the PVP/CA/AE fiber XRD test results graph. The diffraction peaks are 14,61°, 24,11°, and 26,35°. After the addition of *Aloe vera* extract, there was a shift in the diffraction peak from 10.88° to 14,61°, the diffraction peak from 26.56° to 26,35° and the diffraction peak from 21.11°to 24,11°. The shift of the diffraction peaks is caused by amorphization during the spinning process (Sriyanti, Marlina, et al., 2020)

The third peaks represent the PVP, CA, and AE peaks of the PVP, CA, and AE composite fiber. This peak shift indicates that the PVP/CA/EA fiber has a semicrystalline phase. The diffraction peaks shift is caused byamorphization during the spinning process. During this transformation process, the solvent molecules evaporate while the polymer molecules remain bonded together and become stronger. The electric field influences this process in electrospinning (Sriyanti, Agustini, et al., 2020)

Aloe vera extract has several ingredients, such as saponins, alkaloids, antioxidants, carbohydrates, anthraquinones, amino acids, flavonoids, lignin, saponins, acemannan, minerals, and vitamins, which are suitable for wound healing (Liang et al., 2020; Shahzad & Ahmed, 2010; Sharma et al., 2014). The addition of *Aloe vera* extracts to the nanofiber composite to see the physicochemical properties of the fiber so that it can be applied as a wound dressing.

CONCLUSION AND SUGGESTION

It has successfully synthesized PVP/CA nanofibers containing *Aloe vera* extract through electrospinning. The parameters used in the spinning process are the flow rate of 0,20/hour, the voltage of 12 kV, the drum speed of 200 rpm, and the distance of the needle tip to the collector is 75 cm. SEM analysis indicates the addition of *Aloe vera* extract to PVP/CA caused the average fiber diameter to decrease. The XRD crystal structure analysis showed that the PVP/CA/AE nanofibers experienced peak shifts and had a semicrystalline phase.

ACKNOWLEDGMENTS

The publication of this article was funded by DIPA of Public Service Agency of Universitas Sriwijaya 2022. SP DIPA-023.17.2.677515/2022, on December 13,2021. In accordance with the Rector's Decree Number: 0111/UN9.3.1/SK/22, on April 28, 2022 and this article was funded by DIPA of Public Service Agency of Universitas Sriwijaya, 2023. SP DIPA-023.17.2.677515/2023, on November 30, 2022. In accordance with the Rector's Decree Number: 0187/UN9.3.1/SK/2023, on April 18, 2023.

AUTHORS CONTRIBUTIONS

MA reviewed the literature and did experiments. IS built and reviewed the literature. LM reviewed the literature and edited the script. All authors read and agree final script.

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