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SYNTHESIS AND CHARACTERISTICS OF POLYACRYLONITRILE (PAN) NANOFIBER MEMBRANE USING ELECTROSPINNING METHOD

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ABSTRACT

A composite of polyacrylonitrile (PAN) nanofiber by electrospinning is prepared. PAN1, PAN2, and PAN3 with a mass ratio 5, 10, and 15 % w/w, were used, respectively. The characterizations included morphology, crystallinity, and functional groups. The scanning electron microscopy (SEM) shows that the PAN nanofiber with 5 % w/w is a small fiber, while those with 10 % w/w and 15 % w/w are medium and large fibers. The average diameters of PAN1, PAN2, and PAN3 were 130, 443, and 2615 nm, respectively. The Fourier-Transform Infrared analysis shows the presence in the nanofiber the aliphatic hydrocarbons, organic compound, and hydrocarbons compound. The XRD study revealed a fair degree of crystallinity state of PAN nanofibers membrane.

Keywords: electrospinning, nanofiber, polyacrylonitrile.

INTRODUCTION

Nanofiber is an interesting research topic in various fields because it has many advantages and a one-dimensional structure with nanometer dimensions [1]. Nanofiber can be used for various applications, for example, drug delivery systems [1, 2], wound dressing [3], air filters [4], water filters [5], tissue engineering [6], and supercapacitor electrodes [7, 8]. Some methods used to produce nanofiber are designing, figuring the synthesis, phase separation, self-assembly, and electrospinning [9, 10]. Electrospinning is the method for generating nano-sized fibers up to micrometers [1]. Fiber morphology perhaps predicted by modifying electrospinning parameters which consist of: (a) solution (viscosity, conductivity, and solution surface tension); (b) process (high voltage, flow rate, and needle tip distance to the collector); and (c) environmental (humidity) [2, 11]. The advantages of the electrospinning method are its ability to produce large-scale nanofiber of the same size, which can be made from a variety of materials, making the process fast and simple, also the immense ratio of surface area to volume [12].

The polymers that can be used in electrospinning are various. They can be natural or synthetic polymers [1]. Some synthetic polymers that have succeeded in forming nanofiber are polyvinylalcohol (PVA), polyvinylpyrrolidone (PVP), cellulose acetate (CA), and polyacrylonitrile (PAN) [1, 2, 10]. The advantages of PAN polymer are its low density, high polymer strength, elasticity, good solvent resistance, and its ability to maintain morphology in the pyrolysis process [13], so PAN nanofiber is widely developed for membrane air filters, water filters, and carbon fiber [13, 14]. Meanwhile, the suitable solvent for dissolving polyacrylonitrile is usually dimethylformamide (DFM) as an organic solvent. DMF organic compound is a polar solvent with a high boiling point which can act as either electrophilic or nucleophilic material [15]. Besides, DMF can play a role in organic, organometallic, chemistry, and catalysts synthesis [15].

In previous research, Gu et al. (2015) have analyzed the morphology and diameter of PAN/Carbon nanofiber

composites with a diameter of 130 - 280 nm [16]. The stability and morphology of PAN and Gelatin nanofiber has been reported by Sabantina et al. (2018). The stability of the nanocomposite is similar to that of pure PAN with temperature stability at 280°C [17]. The morphology and diameter of polyacrylonitrile (PAN) and crown ether composite nanofibers for selective adsorption, increased with the addition of DB18C6 concentration [18]. However, few PAN nanofiber with analysis of the diameter and crystallinity structure have been reported.

In this report, we used the electrospinning method to synthesize and to characterize the polyacrylonitrile (PAN). The morphology and crystallinity structure of PAN1, PAN2, and PAN3 nanofibers were interpreted by SEM, and X-Ray Diffraction of the nanofibers. The effect of increased PAN concentration nanofibers on the morphology, diameter, crystallinity structure nanofibers, was evaluated.

EXPERIMENTAL

Materials

The material used to make PAN nanofiber is polyacrylonitrile (PAN) with an MW of 150,000 kg mol^{-1} from Sigma Aldrich. The solvent used was dimethylformamide (DMF) gain from Bratacham, Palembang, Indonesia.

Procedure

The process of making a PAN/DMF solution was by dissolving polyacrylonitrile into dimethylformamide with a variety of weight concentrations, i.e. 5 % w/w (PAN1), 10 % w/w (PAN2), and 15 % w/w (PAN3). The polymer solution was stirred with a magnetic stirrer with a rotating speed of 500 rpm for 2 hours at 60°C. The result was then transferred to a 10 ml Terumo® injector with an inner diameter of 15 mm, a needle length of 38 mm, and a 0.8 mm needle diameter.

The electrospinning process is illustrated in Fig. 1. The setup of electrospinning is a syringe pump, rotating collector, high-voltage, and syringe with a needle diameter of 0.81 mm. The temperature and a relative humidity were used for electrospun of nanofibers at 26 ± 0.55°C and 40 ± 50 %, respectively. Firstly, the PAN solution was filled into the syringe. Then, it was distributed by the syringe pump at a constant flow rate of 3 mL h^{-1}. The needle tip was given a positive charge at 14 kV to remove the PAN solution from the needle.

Characterization of Nanofibers

The morphology of the PAN1, PAN2, and PAN3 precursor solutions was examined using an SEM (Tescan, Vega 3). The conductive layer of the sample was applied before SEM imaging. SEM images of the mats were observed at the voltage of 15 kV and 10.000-times optical magnification. Origin ver.8 software was used to determine the size distribution of the fibers. Typical peaks of PAN nanofibers were obtained by Infrared Fourier-Transform Analysis (FTIR). The FTIR spectrophotometer (Bruker, Alpha) with a spectral range of 500 - 4000 cm^{-1} resulted in the FTIR spectra of PAN nanofibers. An X-ray diffractometer (Rigaku MiniFlex, Germany) was also used to obtain X-ray diffraction (XRD) patterns of

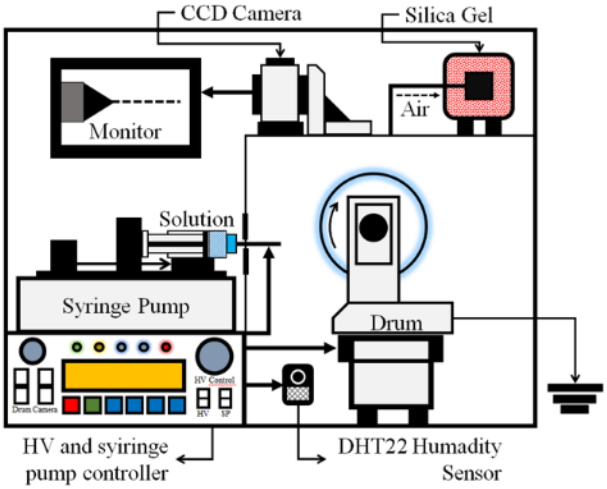


Fig. 1. The scheme of the electrospinning apparatus [1, 2, 19].

PAN1, PAN2, and PAN3 nanofibers. The sample was measured with a wavelength of 1.5405 Å at the voltage and the electric current of 35 kV and 30 mA. The angular position (2θ) was in the range of 5 to 80°.

RESULTS AND DISCUSSION

PAN nanofibers made from the electrospun are shown in Fig. 2. PAN nanofibers have a homogeneous and smooth surface. A successful formation of nanofiber happens when a positive electrode on a needle is connected to a negative electrode at a DC power source so that the initially charged polymer solution is polarized because of the influence of the two potential electrode differences [10, 20]. The Coulomb force is created due to the gathering of these positive. This factor causes the formation of the Taylor cone due to the pull of the polymer solution. A high voltage is applied so that the polymer will experience elongation and the solvent evaporates. The polymer solution will be attracted and emitted in a very small size when this force exceeds the surface tension force of the solution so that the charged polymer solution will be accelerated by the electric field towards the collector. A high voltage is applied so that the polymer will experience elongation and the solvent evaporates. When all is done, the fibers are in solid form on the collector surface [10, 21].

Morphology and Diameter of PAN Nanofibers

The morphology of PAN1, PAN2, and PAN3 nanofibers are shown in Fig 3. In general, the three fibers formed to appear as continuous, flawless ribbon strands such as beads formation, with diameters in the range of nanometer. The difference in PAN concentration does not affect the morphology. The difference in PAN concentration did not affect the shape of the nanofibers and nanofibers were produced in a homogenous form, this finding is consistent with the report of the previous research [20]. Consequently, The concentration in the PAN solution in the electrospinning process has an impact on the formation of PAN nanofibers.

The diameters of PAN1, PAN2, and PAN3 nanofibers vary in the range of 130 to 2615 nm. The average

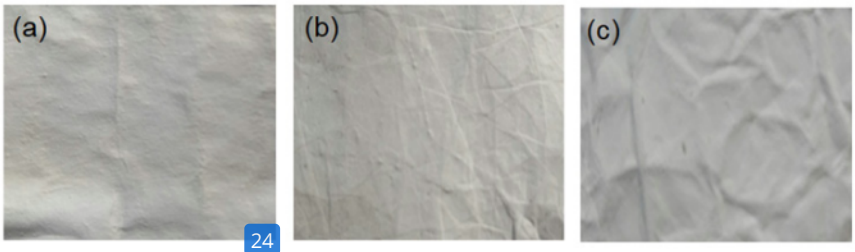


Fig. 2. (a) PAN1, (b) PAN2, and (c) PAN3 nanofibers.

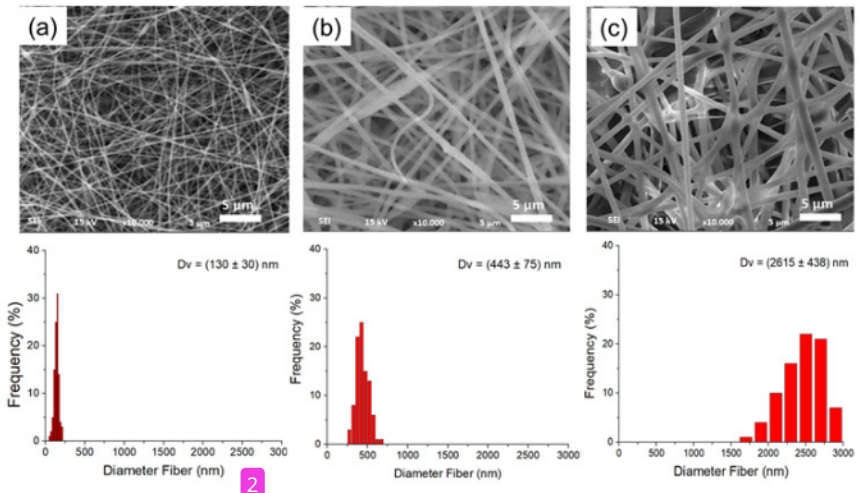
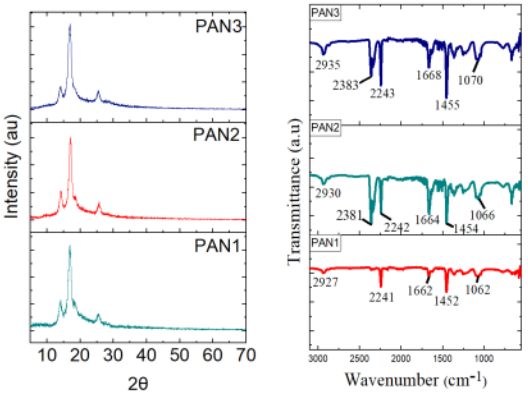


Fig. 3. Morphology and diameter of (a) PAN1, (b) PAN2, and (c) PAN3 nanofibers.



(a) (b)

Fig. 4. (a) X-Ray Diffraction of PAN nanofibers and (b) FTIR spectra of PAN nanofibers.

diameters of PAN1, PAN2, and PAN3 nanofibers are 130 nm, 443 nm, and 2615 nm, respectively. Fig. 3 illustrates the average PAN diameter with three different PAN concentrations. As clearly illustrated in the graph, the diameters of the nanofibers increase along with the increase in PAN concentration. The potential reason for this is the effect of decreasing viscosity [1, 20]. At higher concentrations, more polymer chains are removed at the time specified for spinning (electrospun) [1]. Furthermore, the solvent vaporization is faster in the polymer solution which has a high concentration. This event limits the extension of the polymer while accelerating polymer compaction, resulting in greater fiber formation. This finding is in line with other studies that report similar observations [2, 20, 22].

X-Ray Diffraction Analysis and Fourier-Transform Infrared (FTIR)

The XRD pattern of PAN1, PAN2, and PAN3 nanofibers is shown in Fig. 4(a). In general, the PAN diffraction pattern shows three sharp peaks at 10 - 30° positions. This pattern indicates a fair degree of crystallinity. The PAN1 nanofiber has three sharp peaks at positions of 12°, 17°, and 26°, while the nanofiber PAN2 also has three sharp peaks at 11°, 17°, and 25°, and PAN3 nanofiber has sharp peaks at 11°, 17°, and 24°. The sharp peak at position 17° is found in all three nanofibers. This refers to the PAN background, which is in line with previous studies [23]. However, other sharp peaks owned by PAN pure crystals are not found in all PAN nanofibers. This finding further explains our idea that electrospinning disrupts the state of pure crystals PAN1, PAN2, and PAN3 nanofibers. When charged polymers travel from the tip of the needle towards the collector, the extension of the polymer chain and solvent vaporization occur simultaneously [2]. Because the express vaporization process does not support the pattern of highly-arranged crystals in PAN, the polymer will be in an amorphous structure. This report is related to the research conducted by Sriyanti et al. (2019) that the electrospinning process converts mangosteen crystals to an amorphous state [2].

FTIR studies were conducted to analyze the functional groups of PAN nanofibers. The FTIR pattern of PAN is exposed in Fig. 4(b). The sharp peaks of PAN1, PAN2, and PAN3 nanofibers at 1662 cm^{-1}, 1664 cm^{-1} and 1668 cm^{-1} show C=C bounds of alkene group, and 2241 cm^{-1}, 2242 cm^{-1}, and 2243 cm^{-1} were determined as C≡C bounds as alky group with remove and reduction intensity [25]. Peak 2927 cm^{-1}, 2930 cm^{-1}, 2935 cm^{-1},1452 cm^{-1}, 1454 cm^{-1}, and 1455 cm^{-1} showed C-H stretching of alkane at strong intensity, while peaks 1062 cm^{-1}, 1066 cm^{-1}, and 1070 cm^{-1} on PAN1, PAN2, and PAN3 nanofibers which is attributed to C-H stretching of ester [25]. The sharp peaks of PAN2 and PAN3 nanofibers at 2381 cm^{-1}, 2383 cm^{-1} show C=O bounds of the alkene group [25]. The Increasing concentration of PAN from 5 % (w/w) to 15 % (w/w) and electrospinning process causes a shift in the wavenumber at alkane group (aliphatic hydrocarbons) and an organic compound to a larger wave number.

This result is in line with XRD analysis, indicating that the electrospinning process disrupts the crystal state of PAN1, PAN2, and PAN3 nanofibers. The charged polymer travel from the needle tip to the collector, the polymer elongating chain and evaporating the solvent at the same time. Hence, the pattern of highly-ordered crystallites in PAN nanofibers is not favored by a rapid evaporation process [20].

CONCLUSIONS

PAN nanofiber was synthesized using electrospun. PAN nanofibers have a homogeneous and smooth surface, three factors that influence the formation of PAN nanofibers are Coulomb force, surface tension force, and thrust force of the syringe pump. SEM results show that fibers formed like continuous band strands

without defects such as bead fiber. The increase of PAN concentration results in larger diameters. The diameters of PAN1, PAN2, and PAN3 nanofibers are 130 nm, 443 nm, and 2615 nm. The results of FTIR characteristics also show that increasing the concentration of PAN in nanofiber, and the electrospinning process cause a shift in PAN peaks, i.e. the alkyne and alkene groups (hydrocarbons compound), alkane group (aliphatic hydrocarbons), and organic compound. The XRD results show that the PAN1, PAN2, and PAN3 nanofibers have a degree of crystallinity.

Acknowledgements

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