

SYNTHESIZE AND CHARACTERIZATION OF PVP/CA AND CHITOSAN NANOFIBER USING ELECTROSPINNING

Rafli Fandu Ramadhani¹, Meuretta Alawiyah Pulungan², Meutia Kamilatun Nuha Ap Idjan³, Radiyati Umi Partan⁴, M Rudi Sanjaya⁵, Jaidan Jauhari⁶, Ida Sriyanti^{7*}

^{1,2} Physics Education, Universitas Sriwijaya, Palembang-Prabumulih Street KM.32, Indralaya 30662, ID, Indonesia

^{3,4} Medicine, Faculty of Medicine, Universitas Sriwijaya, Indralaya 30662, ID Indonesia

^{5,6,7*} Electronic and Nanotechnology Applications Research Group, Faculty of Computer Science, Universitas Sriwijaya, Palembang, Indonesia.

Email: ^{7*} ida_sriyanti@unsri.ac.id

*Corresponding author

Abstract

Nanofibers is one of the results of composites that have an important role in the industrial field, implant material, wound dressing, drug delivery and tissue engineering. Manufacturing of nanofibers can be carried out using electrospinning. Electrospinning is an easy, fast and simple technique for produced fibers with sizes ranging from micrometers to nanometers. The purpose of this study were synthesise and characterization of PVP/CA and chitosan nanofiber. Process parameter of electrospinning that used in this research such us flowrate 0,20 ml/hour, high voltage 12 kV, and speed of drum is 200 rpm with the distances to needle tip of collector is 75 cm. The result showed that: (a) Coulomb's law affects the formation of nanofibers. The nanofibers formed are fine, homogeneous and elastic fibers; (b) Viscosity and conductivity affect the diameter of the fiber formed. The average diameter of PCC1, PCC2 and PCC3 fibers are 234 nm, 267nm, and 325 nm. (c) The XRD result shown that the fibers has an amorf phase. The highest sharp peak size of the crystal at angle $2\theta = 22.50^\circ$, crystal plane [h k l] is [2 0 0], and the structures of membrane nanofibers are simple cubic; and (d) The FTIR result shown there are a strong absorption at the O-H bond strain and CH symmetry strain through hydrogen bonds. The fibers were potentially used in implant conductive material.

Keywords: Electrospinning; Morphology; Nanotechnology; Polyvinylpirrolidone

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INTRODUCTION (10%)

Composites are materials made of two or more components with different physical, chemical and mechanical properties. When the components are combined, the composite has better properties than the individual components (S. Jiang et al., 2018). One of the results of composites that have an important role in the industrial field, wound dressing, drug delivery and tissue engineering is fiber (Li et al., 2012; Sriyanti et al., 2017). Manufacturing 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). Electrospinning is an easy, fast and simple technique for producing fibers with sizes ranging from micrometers to nanometers (Gu et al., 2009; Jauhari et al., 2019). Nanofibers produced by electrospinning have advantages such as high

surface area and porosity with small pore sizes, the resulting fibers are very long and continuous and can mimic the extracellular matrix (ECM) thereby increasing cell migration and proliferation (Gu et al., 2009; Nuryantini et al., 2014).

The electrospinning technique involves an electrohydrodynamic process, in which liquid polymer droplets are electrified to produce jets which elongate to produce fibers. During the electrospinning process, liquid polymer is extruded from the injection pump to form Taylor cone-shaped droplets as a result of surface tension (Xue et al., 2018). The components for electrospinning include a syringe, a syringe pump, a spinning nozzle connected to a high-voltage DC, a fiber collector, and a high-voltage power supply (Almafie et al., 2020; Miao et al., 2010).

Electrospinning of abundant and renewable natural biopolymers, such as cellulose and chitin, is becoming an increasingly active research area (Miao et al., 2010). In this study, Polyvinylpyrrolidone/Celulosa Acetate and Chitosan polymers were used. PVP has been widely used in the medical field such as drug release, tissue engineering and wound dressing (Dai et al., 2012; Jauhari et al., 2020; Sriyanti et al., 2021; Virginia et al., 2020). PVP has advantages including biocompatibility, having a structure that can interact with hydrogen bonds, having high adhesion, non-toxicity, excellent electro-feasibility properties, high surface activity and strong adsorption capacity (Bonan et al., 2015; Jauhari et al., 2019; Kurakula & Rao, 2020; Pusporini et al., 2018). However, PVP is easily soluble in water (hydrophilic) which results in reducing the pore size of the membrane thereby reducing the membrane permeans (Erukhimovich & de la Cruz, 2004; Marbelia et al., 2019; Qin et al., 2005). To increase water permeability PVC is combined with cellulose acetate (CA) polymer. Cellulose acetate is insoluble in water. In addition, CA has other advantages such as having high tensile strength, elasticity, heat resistance, low water absorption, easily degraded naturally, biodegradability, biocompatibility, non-toxic, high affinity, good hydrolytic stability, relatively low cost and excellent chemical resistance (Alim Bahmid et al., 2014; Elsayed et al., 2020; Khoshnevisan et al., 2018; Konwarh et al., 2013).

The addition of Chitosan is considered important because chitosan or what is known as chitin is a natural polymer that has advantages such as biocompatibility, biodegradability and low toxicity, properties that make it widely used in agriculture, environmental protection, and biomedicine such as wound healing and antimicrobial agents (Anisie et al., 2023; Bayat et al., 2019; Qiu et al., 2020; J. Wang & Zhuang, 2022). However, producing chitosan nanofibers by electrospinning is not easy because of their high viscosity at low concentrations. Mixing chitosan with different polymers and dissolved in hydrochloric acid or acetic acid functions to form a viscous solution so that this mixture makes the nanofiber bead free with improved mechanical, thermal and structural properties (Nada et al., 2019; Qiu et al., 2020).

Previous research had been carried out using PVP/CA containing *Chromolaena Odorata* extract by (Sriyanti et al., 2021) which showed that the resulting nanocomposite fibers could be applied in the pharmaceutical industry as a potential antioxidant and antibacterial product. Furthermore, similar research is still being carried out by (Sriyanti et al., 2017) using the polyvinylpyrrolidone/cellulose acetate polymer containing *Garcinia mangostana* L extract which is used in the medical field, namely as a drug delivery application. Subsequent research on PVP/Chitosan was conducted by (Zhang et al., 2022) using Chitosan/PVP/Dihydroquercetin and found that films made from these composites were effective in wound healing. However, there has been little research on PVP/CA/Chitosan so far.

Implantable electronic materials for sensing biological phenomena have been widely researched. Materials with high biocompatibility and conductivity have been developed to improve the growth of electrically sensitive tissues, especially nerve tissues and electrically active substrates can promote the functional expression of cellular nerves (W. Wang et al., 2022). To match the electroactive requirements of implanted materials, conductive nanomaterials or electroactive polymers have been usually compounded (Peng et al., 2019). Natural polysaccharide hydrogels typically like chitosan with high biocompatibility and degradability, has been widely used as an implantable material. In this research we will synthesize PVP/CA and chitosan using electrospinning method for produce nanofibers for implantable materials. Further characterization includes SEM, XRD and FTIR tests. The results of the characterization were evaluated to determine the efficacy of PVP/CA and Chitosan nanofiber membranes to produce effective nanofibers for implantable electronics materials.

METHODS (15%)

The materials are PVP with an average molecular mass (Mw) of 1,300,000 produced by Sigma Aldrich, cellulose acetate (CA) with an average molecular mass (Mw) of 30,000 produced by Sigma Aldrich, Chitosan with an average molecular mass of 50,000 produced by production by Sigma Aldrich and acetic acid obtained from Brataco Chemical.

Precursor solutions were prepared by dissolving PVP and CA in acetic acid. The PVP/CA solution was prepared at a concentration of 10.4% (w/w) with a ratio between polymer and solvent of 7:3 and stirred for 2 hours. Next, added Chitosan into a homogeneous PVP/CA solution with a concentration ratio of 0.5%;1%;1.5% (w/w) labeled with PCC1, PCC2 and PCC3 and stirred using a magnetic stirrer for 2 hours. The precursor solution was put into the syringe to be spun for 7 hours with flow path parameters of 0.20 ml/hour, voltage of 12 kV, drum speed of 200 rpm and distance of needle tip to collector of 75 cm. PVP/CA/Chitosan nanofibers are produced using an electrospinning apparatus.

The morphology of nanofibres from PVP/CA/Chitosan solutions was characterized using a Scanning Electron Microscope (SEM), the average size of fiber diameter was analyzed using ImageJ 1.52a software, the size distribution of nanofibers was characterized using Originpro 2018 software. Furthermore, functional groups and molecular interactions in PVP/CA/Chitosan were identified using Fourier Transform Infra Red (FTIR). The average size of crystallization or crystallinity in PVP/CA/Chitosan uses X-Ray Diffraction (XRD). The results of the data from this experiment were then analyzed in order to get a conclusion.

RESULTS AND DISCUSSION (70%)

A. Effect of Coulomb Law on the electrospinning process in the formation of fibers.

One of the techniques in producing nanofibers is through the electrospinning technique. Electrospinning can produce nanofiber-based polymers that have high performance and functionality (Shukry et al., 2014). The mechanism in the electrospinning process starts from adding the polymer solution into the injector. Furthermore, the injector is connected to a booster pump. Then, a DC voltage source is connected to the syringe which as the positive electrode while the drum as the negative electrode. When the two electrodes are given a high voltage source, the polymer solution which is initially neutral will experience polarization because there

is a potential difference between the two electrodes. If the needle is connected to the positive pole of the DC voltage source, positive ions will collect on the surface of the solution, while negative ions will collect in the middle. The polymer liquid is extruded from the injection pump to form Taylor cone as a result of this surface tension which becomes nanofibers (Xue et al., 2018).

The positively charged polymer solution connected to the negative electrode of the drum creates an external electric field between the tip of the syringe and the collector. This electric field serves to control the jet charge that comes out of the syringe. The magnitude of the electric force is:

$$F_E = qE \quad (1)$$

q is the electric charge (C) and E is the electric field strength (N/C). The interaction force between two charges q is known as the Coulomb force, namely:

$$F_C = k \frac{q_1 q_2}{r^2} \quad (2)$$

These forces cause the polymer solution to be attracted, the resultant of the two forces exceeds the force due to surface tension and the bonds between the polymer chains which work against the electric and Coulomb forces, the charged polymer solution will be accelerated towards the drum by the electric field. The stretched polymer solution elongates and the solvent evaporates under the influence of the electric voltage. This is what causes the polymer solution to form on the surface of the drum in the form of fibers. The fibers produced through the electrospinning process are in the form of fine, homogeneous and elastic fibers. The result can be seen in the image below.



Figure 1. Nanofibers of (a) PCC1, (b) PCC2, (c) PCC3

B. Effect of Viscosity and Conductivity on the morphology of PVP/CA and Chitosan nanofiber

To see the morphology of PVP/CA and Chitosan fiber used Scanning Electron Microscope. The fiber diameter distribution of PVP/CA and Chitosan is in the range of 160 nm - 440 nm. The average diameter of PCC1, PCC2 and PCC3 fibers respectively are 234 nm; 267nm; 325 nm, with a standard deviation of 58 nm; 48nm; 65 nm and the coefficient of variation is 0.90; 0.98; 0.96. PCC1 fiber with a Chitosan concentration of 1.5% produces bead fibers. Where as for PCC2 and PCC3 with chitosan concentrations of 1% and 0.5% produced fine fibers that were bead free and continuous.

The addition of chitosan causes a difference in morphology. Fiber with a chitosan concentration of 1.5% shows bead fiber. This is due to the increase in the viscosity of the solution on the fiber so that the amount of polymer mass to volume increases and causes the average diameter of the fiber to increase. The lower

concentration of chitosan, bead structure in the fiber decreases, and the fine fiber increases. Viscosity and conductivity affect each fiber diameter. Viscosity has a direct relationship with the molecular mass and concentration of the polymer solution. If the viscosity is higher, the polymer molecule bond will be stronger. In theory, it is expected that the lowest concentration of polymer would produce the smallest diameter fiber (Grant et al., 2021). The highest concentration of polymer often suggests the highest average fiber diameter but this is not the case in this experiment. This is in line with research conducted by J Grant (2021) The previous literature has suggested a non-linear relationship between solution concentration and fiber diameter. It may causes by the beaded fiber. Reducing the concentration of chitosan causes a decrease in the conductivity of fiber. The lower the conductivity value, the less charge accumulated in the solution so that the elongation decreases and causes the average diameter of the fiber to increase.

Then, the size distribution of PVP/CA and Chitosan fibers is shown in Figure 2. Based on the graph, it can be seen that there is an increase in the average fiber diameter when the concentration of chitosan decrease to the PVP/CA matrix. In this study, Reducing the concentration of chitosan causes the viscosity value of the polymer to increase which causes the polymer molecule bonds to become stronger. by decreasing the concentration of chitosan, the bead structure on the fiber is reduced (L. Wang et al., 2019).The morphology from PVP/CA/Chitosan nanofibers is showed in figure 2.

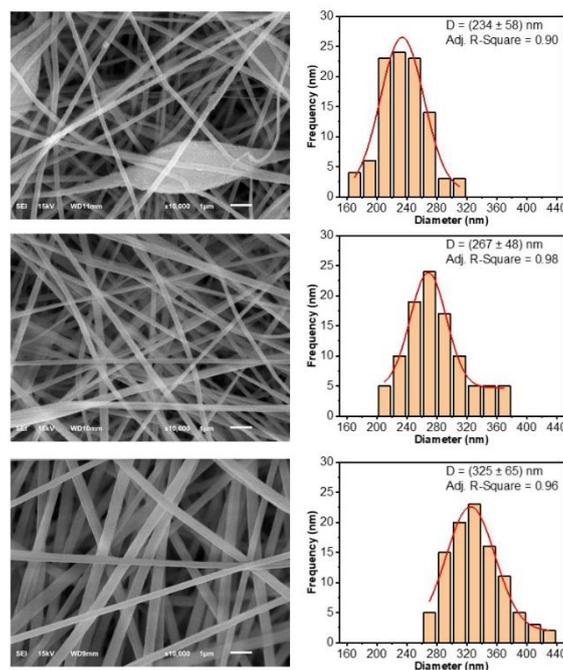


Figure 2. SEM and diameter distribution of nanofiber PCC1;PCC2;PCC3 nanofiber

(c) Effect of electrospinning process on the crystallinity structure of PVP/CA and Chitosan nanofibers

Crystallinity analysis on PVP/CA and Chitosan using XRD test. The XRD patterns of PVP/CA and chitosan are shown in Figure 3. Figure 3 (a-c) represents PVP/CA and chitosan with different chitosan concentrations. For (a) PCC1 nanofiber

has three sharp diffraction peaks between angles 2θ at 5° to 40° , namely 10.78° , 20.16° , 22.18° . Furthermore, (b) PCC2 nanofiber has diffraction peaks at 10.81° , 20.12° , 22.22° . (c) PCC3 nanofiber has three sharp diffraction peaks of 10.68° , 20.22° , 22.50° . The XRD results of PCC1, PCC2, and PCC3 nanofiber indicate an amorphous phase. This is in accordance with previous research conducted by Sriyanti et al (2018) that PVP and CA have an amorphous phase.

Pure chitosan has a crystalline phase however, when chitosan is combined with other polymers then chitosan turns amorphous. This is in line with research conducted by Qi 2004 which states that there are two peaks on the XRD of pure chitosan but no crystalline peaks were found on chitosan nanofibers membrane. Amorphization occurs because when the solution is drawn towards the collector, the liquid turns into a solid. During this transformation process, the solvent molecules evaporate while the polymer molecules remain bound and become stronger. This process is influenced by the electric field in electrospinning (Sriyanti dkk, 2018).

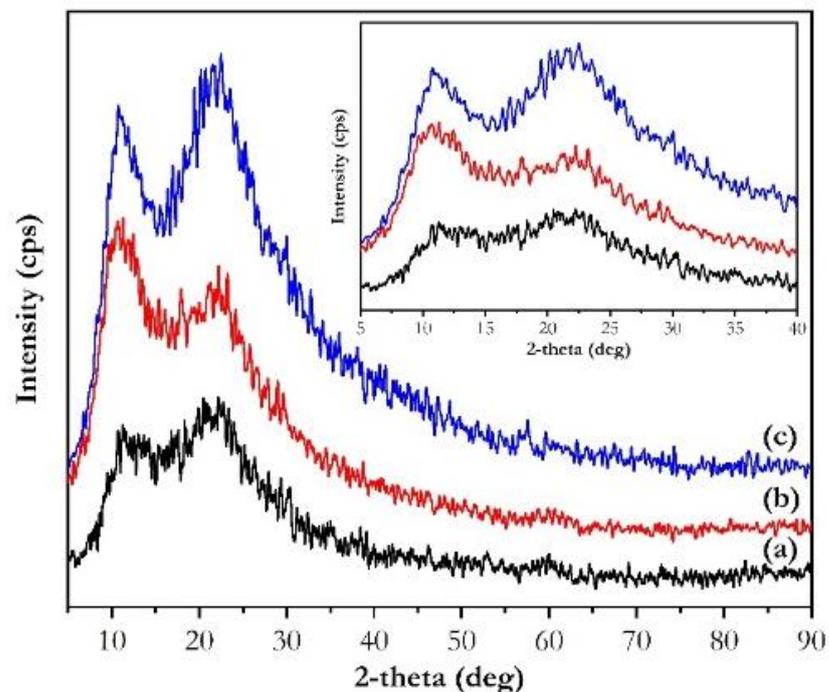


Figure 3. XRD result of PCC1, PCC2, and PCC3 nanofiber

We also analyzed the crystal size or amorphous of nanofiber membrane. The size of crystal or amorphous can be calculated using Scherrer equation show in equ (3):

$$d = \frac{k\lambda}{\beta \cos \theta} \dots \dots \dots (3)$$

Where d is the crystal (amorphous) size, β = Full width at half maximum, λ is the wavelength of X-rays (Cu-K α : $\lambda = 1.5406 \text{ \AA}$), k is the Scherrer constant (k) which has a price of 0.9. The crystal size of PCC1, PCC2, PCC3 show in Tabel 1. The highest sharp peak size of the crystal at angle $2\theta = 22.50 \text{ \AA}$. while the crystal plane represented by Miller indices $[h \ k \ l]$ of PCC1, PCC2, PCC3 nanofibers are $[2 \ 0 \ 0]$. This crystal plane was found in accordance with previous research report (L. Jiang et al, 2020), where this crystal plane $[h \ k \ l]$ of PVP/CA is $[2 \ 0 \ 0]$ with diffraction angle of 22° . Crystal plane images of PCC1, PCC2, and PCC3 are shown in Figure 4. From

the analysis of the crystal plane images, it can be concluded that the structures of PCC1, PCC2, and PCC3 nanofibers are simple cubic.

Table 1. Crystal Size of PCC1, PCC2, and PCC3 nanofiber

Sample	2θ ($^\circ$)	β ($^\circ$)	Crystal Size (\AA)
PCC1	10.78	12.6	3.88
PCC2	22.18	5.3	7.69
PCC3	20.16	5.5	4.00

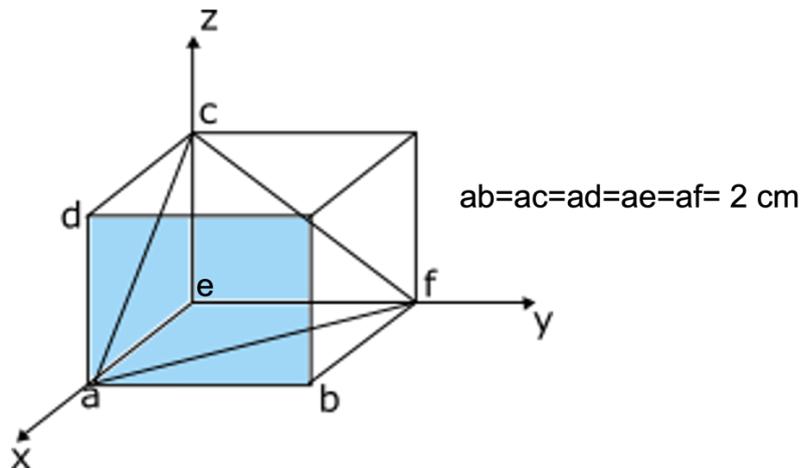


Figure 4. Crystal Plane Images of nanofiber

D. Effect of FTIR test of PVP/CA/Chitosan Nanofibers

The results of the nanofiber FTIR test are shown in figure 4 below. In this FTIR test using a wave number of $550 - 3850 \text{ cm}^{-1}$.

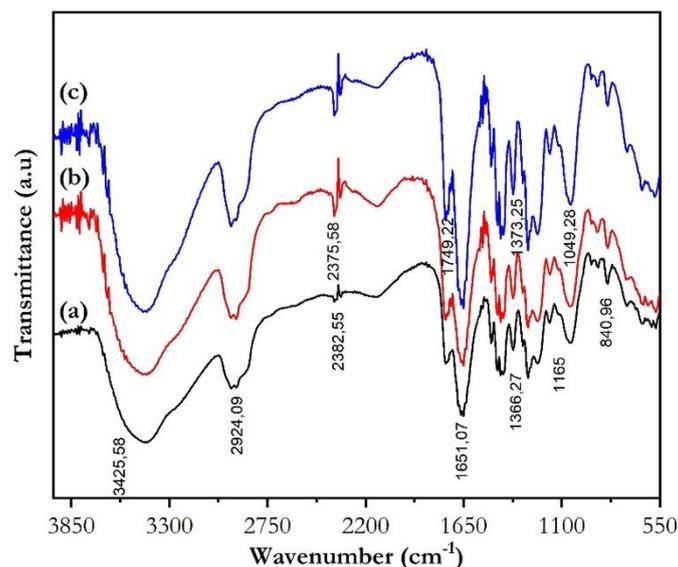


Figure 5. FTIR result of PCC1;PCC2;PCC3 nanofiber

Based on Figure 5 (a-c) represents the FTIR results of PCC1, PCC2 and PCC3 fibers sequentially with varying concentrations of Chitosan. FTIR characterization results on PCC1 fiber characteristic peaks can be observed at wave number 3425,58

cm^{-1} , $2924,09 \text{ cm}^{-1}$, $2382,55 \text{ cm}^{-1}$, $1651,07 \text{ cm}^{-1}$, $1366,27 \text{ cm}^{-1}$, 1165 cm^{-1} and $840,96 \text{ cm}^{-1}$, PCC2 the characteristic peak is observed at wave number $3425,58 \text{ cm}^{-1}$, $2924,09 \text{ cm}^{-1}$, $2375,58 \text{ cm}^{-1}$, $1651,07 \text{ cm}^{-1}$, $1366,27 \text{ cm}^{-1}$, 1165 cm^{-1} and $840,96 \text{ cm}^{-1}$. Then, for PCC3 it has a peak in wave number $3425,8 \text{ cm}^{-1}$, $2924,09 \text{ cm}^{-1}$, $2375,58 \text{ cm}^{-1}$, $1749,22 \text{ cm}^{-1}$, $1373,75 \text{ cm}^{-1}$, $1049,28 \text{ cm}^{-1}$ and $840,96 \text{ cm}^{-1}$.

In general, the chitosan saccharide wave band is in the range of $1000\text{--}1200 \text{ cm}^{-1}$ (Grant et al., 2021) From the test results on PCC1-PCC3 fibers it is shown that the wave numbers are 1165 cm^{-1} and 1049 cm^{-1} . This wave number indicates a strong absorption in the C-O bond. The presence of PVP in PCC1, PCC2 and PCC3 fibers is marked with a wide band of 3425.58 cm^{-1} indicating the O-H stretch of the hydroxyl group (Prayuddy et al., 2017) While the wave number 2924.09 cm^{-1} represents the CH symmetry strain. This is in line with research by (Paipitak et al., 2011) which said that the OH absorption band was in the range $3400\text{--}3500 \text{ cm}^{-1}$ and the wide band for CH absorption was in the range $2900\text{--}3000 \text{ cm}^{-1}$. The presence of acetyl groups from cellulose acetate is evidenced by the wave numbers at 1651.07 cm^{-1} , 1288.45 cm^{-1} , 1234.44 cm^{-1} indicating the presence of C=O and C-O bond vibrations.

The decrease in chitosan concentration caused a shift in the peak of Chitosan at 1165 cm^{-1} to 1049 cm^{-1} . The reduction of chitosan causes a change in the characteristic peaks of the wave number. Changes in the peaks of wave numbers are caused by molecular interactions in the polyvinylpyrrolidone and cellulose acetate polymers with molecules in chitosan in the form of hydrogen bonds.

CONCLUSION (5%)

PVP/CA/Chitosan nanofiber has been successfully synthesized by electrospinning method. The parameters used in the spinning process were a flow rate of 0.20/hour, a voltage of 12 kV, a drum speed of 200 rpm and a distance of 75 cm from the needle tip to the collector. Formation of fibers influenced by Coulomb Law. SEM nanofiber analysis showed that the addition of chitosan caused a difference in the average fiber diameter of PCC1, PCC2 and PCC3 experienced. when the concentration of chitosan decrease the average fiber diameter was increase because of the viscosity of solution. The results of crystal structure analysis using XRD showed that the PVP/CA and Chitosan nanofiber is amorphous phase. The highest sharp peak size of the crystal (amorphous) at angle $2\theta = 22.50 \text{ \AA}$, crystal plane [h k l] is [2 0 0], and the structures of PCC1, PCC2, and PCC3 nanofibers are simple cubic. The results of chemical interaction shows that PCC1, PCC2, PCC3 there are a strong absorption at the O-H bond strain and CH symmetry strain. PCC1, PCC2, and PCC3 nanofibers have potential as conductive material for cardiac implant applications

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